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U. S. DEPARTMENT OF AGRICULTURE

DIVISION OF CHEMISTRY

BULLETIN No. 31

PROCEEDINGS

OF THE

EIGHTH ANNUAL CONVENTION

OF THE

ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS

HELD AT

THE COLUMBIAN UNIVERSITY, WASHINGTON, D. C.

AUGUST 13, 14, AND 15, 1891

METHODS OF ANALYSIS OF COMMERCIAL FERTILIZERS, FOODS AND FEEDING STUFFS, DAIRY PRODUCTS, FERMENTED LIQUORS, AND SUGARS

EDITED BY

HARVEY W. WILEY

SECRETARY OF THE ASSOCIATION

PUBLISHED BY AUTHORITY OF THE SECRETARY OF AGRICULTURE

WASHINGTON

GOVERNMENT PRINTING OFFICE

1891

LETTER OF TRANSMITTAL.

U. S. DEPARTMENT OF AGRICULTURE,
DIVISION OF CHEMISTRY,
Washington, D. C., November 21, 1891.

SIR: I have the honor to submit the report of the Proceedings of the Eighth Annual Convention of the Association of Official Agricultural Chemists, with the recommendation that it be published, as heretofore, as a bulletin of the Chemical Division of the Department.

Respectfully,

H. W. WILEY,
Chemist.

HON. J. M. RUSK,
Secretary of Agriculture.

PROCEEDINGS OF THE EIGHTH ANNUAL CONVENTION OF THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS, HELD AT WASHINGTON AUGUST 13, 14, AND 15, 1891.

FIRST DAY.

MORNING SESSION, THURSDAY.

In accordance with the call of the executive committee, the association met in the lecture hall of the Columbian University at 10 o'clock, the president, Mr. G. C. Caldwell, in the chair.

The following members and "others interested in the objects of the association" were present:

Hon. Edwin Willits, Assistant Secretary of Agriculture.

The president, Mr. G. C. Caldwell, chemist of the New York Agricultural Experiment Station, Ithaca, N. Y.

The vice-president, Mr. N. T. Lupton, chemist of the Alabama Experiment Station, and official chemist of the Alabama State Department of Agriculture, Auburn, Ala.

The secretary, Mr. H. W. Wiley, chief chemist, U. S. Department of Agriculture.

Members of the executive committee, Mr. E. B. Voorhees, of New Brunswick, N. J., and Mr. R. C. Kedzie, of Agricultural College, Mich., and the following members of the association and participants:

Adriance, Duncan, College Station, Tex.
Allen, E. W., Washington, D. C.
Anderson, James T., Auburn, Ala.
Babcock, S. M., Madison, Wis.
Bartlett, J. M., Orono, Me.
Battle, H. B., Raleigh, N. C.
Beal, W. H., Washington, D. C.
Carpenter, F. B, Raleigh, N. C.
Cathcart, Chas. S., New Brunswick, N. J.
Clarke, F. W., Washington, D. C.
Collingwood, Charles B., Tucson, Ariz.
Cooke, W. W., Burlington, Vt.
Crampton, C. A., Washington, D. C.
Crawley, J. T., Audubon Park, New Orleans, La.
Dabney, Chas. W., Knoxville, Tenn.
Davidson, R. J., Blacksburg, Va.
Davis, I. T., Washington, D. C.
De Roode, Rudolf, Morgantown, W. Va.
Edson, Hubert, Patterson, La.
Elliott, J. C., Rittman, Ohio.
Farrington, E. H., Champaign, Ill.
Fellows, G. S., Washington, D. C.
Frear, William, State College, Pennsylvania.
Fristoe, E. T., Washington, D. C.
Fuelling, Jno. L., Washington, D. C.
Gaines, Richard H., Richmond, Va.
Gascoyne, W. J., Baltimore, Md.
Harper, D. N., Minneapolis, Minn.

Harrington, H. H., College Station, Tex.
Herff, B. v., Washington, D. C.
Horton, Horace E. L., Audubon Park, New Orleans, La.
Huston, H. A., La Fayette, Ind.
Jenkins, E. H., New Haven, Conn.
Johnson, D. D., Morgantown, W. Va.
Jordan, Whitman H., Orono, Me.
Kilgore, B. W., Raleigh, N. C.
Knorr, Aug. E., Washington, D. C.
Krug, William H., Washington, D. C.
Lord, N. W., Columbus, Ohio.
Maxwell, Walter, Washington, D. C.
McAdie, Alexander George, Washington, D. C.
McElroy, K. P., Washington, D. C.
McDonnell, H. B., College Park, Md.
Morse, Fred. W., Hanover, N. H.
Myers, John A., Morgantown, W. Va.
Nicholson, H. H., Lincoln, Nebr.
Ogden, A. W., New Haven, Conn.
Patrick, G. E., Ames, Iowa.
Patterson, Harry J., College Park, Md.
Patterson, L. G., Agricultural College, Mississippi.
Payne, George F., Atlanta, Ga.
Penny, C. L., Newark, Del.
Robinson, Norman, Tallahassee, Fla.
Robinson, S. A., Orlando, Fla.
Ross, B. B., Baton Rouge, La.
Schweinitz, E. A. v., Washington, D. C.
Scovell, M. A., Lexington, Ky.
Shepard, Jas. H., Brookings, S. Dak.
Street, John P., New Brunswick, N. J.
Stubbs, W. C., Audubon Park, New Orleans, La.
Sutton, D. C., Narcoossee, Fla.
Teller, George L., Fayetteville, Ark.
Terne, Bruno, Philadelphia, Pa.
Van Slyke, L. L., Geneva, N. Y.
Voorhees, Louis A., New Brunswick, N. J.
Wheeler, H. J., Kingston, R. I.
Wilkinson, L. W., Auburn, Ala.
Williams, W. J., Camden, N. J.
Winton, A. L., New Haven, Conn.
Withers, W. A., Raleigh, N. C.
Woods, Charles D., Middletown, Conn.

Mr. R. Warington, of St. Albans, Herts, England, was also present, and was appropriately greeted by the president as the representative of the celebrated Agricultural Experiment Station at Rothamstead.

Mr. Wiley presented the following report of the executive committee on the order of business:

PROGRAMME.

Hours of Meeting.—Morning hours, 9:30 to 12:30; afternoons, at 2.

1. Address of the president.
2. Report on analysis of fermented liquors.
3. Report on analysis of dairy products.
4. Report on analysis of foods rich in carbohydrates.
5. Report on analysis of foods poor in carbohydrates.
6. Report on analysis of sugars.
7. Miscellaneous papers and business; election of officers, etc.
8. Report on determination of nitrogen.
9. Report on determination of potash.
10. Report on determination of phosphoric acid.
11. Report on analysis of ash and soil.

The committee recommends that immediately following the reading of each report any written communications relating to the subject of such report shall be presented and read, the discussion upon the report to be deferred until such papers, if any, have been submitted to the association.

On motion, the report of the committee was accepted, and the order of business therein recommended was adopted.

ADDRESS OF MR. G. C. CALDWELL, PRESIDENT.

The president addressed the association as follows:

GENTLEMEN OF THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS: The fallibility of methods of chemical analysis is especially exposed so soon as they come to be rigorously tested by many analysts working in concert, and the more such testing of methods come to be applied, the clearer is the demonstration of the necessity for just the kind of work that this association has been engaged in now for eight years. In casting about for a subject on which to address you, in accordance with the established custom, at the opening of this, the eighth annual session of the association, over which you have done me the honor of calling me to preside, it occurred to me that a brief review of the history and present condition of efforts for the establishment of uniformity in methods of technical chemical analysis might be both interesting and useful, and perhaps also pleasantly self-exalting, if it should show that we occupy an advanced position in this respect.

To the agricultural chemists of German experiment stations appears to belong the honor of first proving the necessity of uniformity in methods of technical analysis, and of testing the methods in common use for the purpose of ascertaining which of them will yield the most uniform results when used by many chemists, and in what ways they may be improved.

In 1872[1] Henneberg, Kühn, Maercker, and two of the Schulzes, chemists especially interested in the system of fertilizer control by the coöperation of the experiment stations, observing such serious discrepancies in the results of determination of phosphoric acid by different analysts, called for a conference on unification of methods of estimation of this constituent of fertilizers. Nineteen chemists, many of them as well known by reputation to all older students of agricultural chemistry as those who issued the call, responded. The most important result of the deliberations was an agreement on the conditions under which the uranium method might be used, and the molybdate method must be resorted to, full directions being given for both methods.

I find no further reference to this subject till 1880, when there were published[2] the results of five analyses of the same fertilizer by several chemists, showing, in particular, a difference of 2 per cent on the reverted acid; fair agreement on the soluble acid, but a difference of half a per cent on nitrogen, and nearly 2 per cent on potash. But no action appears to have been taken then in regard to the matter.

In 1881[3] the most important of the early conferences on this subject was held. It was participated in by sixty-four experiment station directors, and agricultural and trade chemists. Henneberg was

[1] Versuchs-Stat., XVI, 233.

[2] Journal d'Agriculture Pratique, 1880, 112.

[3] Journal für Landwirthschaft XXX, 157.

chairman of the meeting, and the names of most of the experiment station workers, well known to us, are to be found in the list of those present.

The deliberations were confined, however, exclusively to the phosphoric acid; this topic was very thoroughly treated; the mode of preparing the sample, extraction of the water-soluble acid, its volumetric estimation by uranium solution, the gravimetric method by molybdate and magnesia mixture, the estimation of the citrate-soluble acid, and of the total insoluble acid, in bone meal and rock phosphate, were discussed and provided for.

Next in order, chronologically, I find in the Chemical News[1] that a comparison of results obtained by several trade chemists on the estimation of water-soluble phosphoric acid in superphosphates disclosed differences no greater than 0.5 per cent, and the conclusion that a conference on unification of methods was unnecessary. Also a discussion took place in the same year in the Country Gentleman, published at Albany, N. Y., in which the reliability of the analyses of superphosphates was attacked and defended. There appeared also in this year[2] a report covering seventy-five pages of a subcommittee of a committee of the agricultural experiment stations of France, on unification of methods of analysis of fertilizers, this committee being composed of Schloesing, chairman; and Girard, Grandeau and Muntz. Very full descriptions are given of methods to be employed, two methods being usually allowed for each substance. For phosphoric acid, magnesia precipitation in citric acid solution was allowed, and the molybdate method is prescribed only when but little acid is present. There is no mention of any concerted work in testing methods. In the same year also, the first session of this association was held at Philadelphia, when the methods of estimation of phosphoric acid and potash were discussed, and action towards unification was taken; but nitrogen was not touched. The report of this meeting was embodied in a pamphlet of eight pages.

In 1886[3] a small side issue arose in the shape of an agreement between the experiment stations at Bonn, Darmstadt, Speyer and Wiesbaden, and the fertilizer manufacturers, whose products were put under their control, by which the phrase soluble phosphoric acid was defined to mean that soluble in solution of ammonium citrate, and precipitated from that solution directly by molybdate.

In September, 1888,[4] at a meeting of the Association of Agricultural Experiment Stations of Germany, methods were reported by a committee, and adopted, for the estimation of phosphoric acid in Thomas slag phosphate, and also for the determination of the fineness of grain, and for the estimation of nitrogen in nitrates.

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[1] XLIX, 239, 1884.

[2] Annales Sc. Agronomique, 1884, 274.

[3] Fresenius, Zeitschrift, XXV, 271.

[4] Versuchs-Stat., XXV, 437.

In November, 1889,[1] a meeting was held at Hanover of chemists of German fertilizer companies, and technical chemists, with O. Gussfeld as chairman, where a long discussion took place on the improvement and extension of the methods of analysis agreed upon at Halle eight years before. In January, 1890,[2] there was a meeting at Leipzig of a local union of chemists of experiment stations, and agents of fertilizer manufacturers, at which only 12 persons were registered as present. Mærcker was chairman, but very few other names are familiar to me. The volumetric uranium method was discussed, and condemned by many. A resolution was offered to abandon it, but not adopted. The report of the meeting at Hanover in the preceding November was taken up and discussed, but no definite conclusions in regard to the same were reached. Proposals and suggestions from the fertilizer manufacturers were received, and a plan was adopted for work on doubtful matters. The analytical questions were to be worked upon till fall, and an exchange of opinions was to be given then, at the meeting of the Association of Experiment Stations in Bremen.

This meeting, at which 40 were registered as present,[3] was presided over by Dr. Nobbe. It was stated that four committees of the association had met during the year; the fertilizer committee met at Leipzig and at Bremen. At both meetings invited representatives of fertilizer manufacturers and technical chemists were present. The fodder analysis committee met at Bernburg, at which representatives of the German agricultural council were present. The soil analysis committee met at Darmstadt.

A scheme for the analysis of fertilizers was reported, discussed at length, and many of its propositions were adopted. Very precise directions were given for the taking and transportation of the sample, and its treatment when received. The conclusion was finally adopted that the molybdate method must be resorted to in all decisive cases. It was decided that, as to the direct precipitation of soluble acid from citrate solution, it shall be tested on samples sent out by a committee, and the results shall be considered at the next meeting of the association; that for the estimation of aluminum and iron in phosphate, Glaser's method is to be strictly followed, provisionally; but no provision was made for testing it; considering the discussion of this method, which is still going on, such is not the kind of action that would have been taken by this association.

In respect of the analysis of fodders, but little in the way of coöperative testing and unification of methods has been done in Germany, and nothing elsewhere, except in this country. The so-called Weende method has been adopted everywhere by tacit consent or special agreement. Such modifications, none of them radical, of the original method

[1] Zeit. Angewand. Ch., 1889, 690, and 1890, 62.

[2] Versuchs-Stat., XXXVII, 291.

[3] Versuchs Stat., XXXVIII, 281.

as have been made were adopted at meetings of experiment station chemists after discussion of individual tests that were reported. Only the ether extract has received any concerted attention in the form of committee tests, and the subsequent discussion and adoption of a method reported. Even at the last meeting of the Association of Experiment Stations, in 1890, this estimation was the only one in regard to which any action was taken prescribing the manner in which it should be conducted, although resolutions were adopted as to further work on fodders on a common plan.

In connection with the Paris Exposition, in 1889, an International Chemical Congress was held, at which methods of analysis of soils, fertilizers, and fodders were adopted.[1] But I do not think that many chemists, other than those present at the meeting, if indeed all of these, will regard themselves as very strictly bound by this indorsement of certain methods. It can hardly be expected that, in the midst of such distractions as would be necessarily incident to the proximity of that great exposition, there could be that careful survey of the whole field, and examination of all the work that has been done on the various methods involved, which would be an essential preliminary to the adoption of certain ones as the best for general acceptance, to the exclusion of others. How widely, for instance, will this method for nitrates in soils meet with such general adoption without preliminary careful testing: "Treat the solution of the soil with lime and alcohol of 50 per cent; sulphates, phosphates, and silicates remain in the residue; evaporate the solution containing the nitrates to dryness with ammonium chloride, thus converting all the former into chloride; estimate chlorine in this residue, as well as in a portion of the original sample, and from the difference calculate the nitrates in the soil."

In respect of the analysis of milk the Society of Public Analysts in England have made some important contributions towards the unification of methods. The special indorsement of the Adams paper coil by this society has given it and other methods based on a similar principle, such as the asbestus method, very wide use, and in so far has probably led to greater uniformity in the results on the estimation of the fat in milk.

In this country, besides our own work, we had last year the report of the committee of the Louisiana Sugar Chemists Association, based on the results of a very careful investigation of different methods of determining sugar, and describing in detail the methods for general adoption.

The Union of Swiss Analytical Chemists adopted in September, 1889, a method for the analysis of wine; a careful definition of terms was also adopted, such as alcohol, extract, acidity, etc. A set of preliminary tests is described, as odor, color, taste, clearness. As to the particular methods of estimation of each constituent, however, we find no records of coöperative testing; even for the description of these methods we

[1] Journal Society Chem. Ind., 1890, 115.

are referred to the original accounts of them in this or that work or journal.

Such are at least the main points in the history of the unification of methods of analysis of agricultural materials and products, outside of our own work. In respect of other branches of technical analysis, I find but little of such effort recorded. In 1883,[1] Lunge, of Zürich, was requested by a committee of the German Society for the Promotion of Chemical Industry to submit to its next general meeting this proposal: "That steps be taken for bringing about an international agreement concerning uniform analytical methods for estimating the commercial value of certain products." The suggestion leading to this action came from a chemical fertilizer manufacturer. In a circular letter sent to leading chemists, Lunge refers to the confusion and lawsuits resulting from the use of different methods of sampling or analyzing, or both; to the desirability of stating results in a uniform manner; to what has been accomplished in respect of the trade in chemical manures; to the efforts of the alkali trade in this direction by the publication of a manual for alkali, potash, and ammonia works, in which methods of testing the crude materials and products of the alkali industry were recommended for general adoption. He allows that no methods of analysis should be held as binding for any long period, and that even with such allowance it would be impracticable to compel all chemists to follow any one method; but he thinks that certain methods, generally agreed upon, might serve as standards of reference in disputed cases, and be acknowledged as decisive in the courts of law.

Finally he addresses six questions to chemists, the most important of which are the following four:

(1) Whether there can be an international agreement concerning methods of sampling technical materials.

(2) The same with reference to methods of testing or assaying the same.

(3) The same with reference to methods of stating results.

(4) Whether methods agreed upon should be made binding for an indefinite time, or whether provision should be made for their revision from time to time, and publication of the results of such action.

Whether the methods adopted at the International Chemical Congress at Paris, above referred to, were the outcome of this action, I have not been able to learn; but I find no further reference to the matter in any of my notes on the subject.

In the same year the Union of German Iron Furnace Men appointed a committee, of which Skalweit was chairman, for the consideration of the unification of methods of analysis of iron and steel. The chairman seems to have been the head and soul of the project, for his death soon after put such a damper on the enterprise that nothing further is mentioned about it till 1888, when a self-appointed committee of chemists

[1] Chemical News, XLVII, 210.

took it up, and sought to excite a more general interest in the matter by sending out a circular of questions to forty-five iron-furnace laboratories. The estimation of manganese was grappled with first, as being the most difficult, and it was decided to study the volumetric methods. One member of the committee, apparently well satisfied with his own knowledge of the subject, withdrew, because he considered gravimetric methods as the only reliable ones, in face of the fact that probably at least three-fourths of the iron chemists use one volumetric method or another. A subcommittee of this original committee met to carry on together the work of testing methods; they were very soon able to show the need of the work they were engaged in. Two volumetric methods were fixed upon as the most reliable, and very precise directions were given for carrying them out.

In the Proceedings of the American Institute of Mining Engineers there are numerous valuable papers on the methods of analysis of iron and steel, and some coöperative work is recorded there, done at the request of some one or another, authors of papers in which the work is described. The need of improvement and unification of methods of analysis was so clearly shown in these papers, and others in foreign periodicals, that one naturally wonders that no concerted action for the attainment of this end was taken till 1888. Then, at the suggestion of one of our own countrymen, Prof. J. W. Langley, an international organization was formed consisting of subcommittees in each country represented in the organization. The object was stated to be the making up of a set of samples of steel, to be analyzed with great care, portions of which should then be put in some place of deposit in each country; these samples should then be recognized as standards, to which scientific and commercial analysis of iron and steel could be referred in cases where questions of accuracy should arise.

The American committee came to this conclusion[1] at its very first meeting: "That the whole subject of the existing methods for the determination of carbon in iron and steel seemed to be affected by so many discrepancies and differences of opinion that, before beginning work on the international standard, it would be desirable to make a preliminary study of methods." Therefore, before proceeding to analyze and fix the composition of the valuable international samples, the committee set out on the line of investigation, in which this association has been a pioneer, in order to fix upon the best method of analysis for final use.

It appears to me that the hope expressed in the beginning of my address, that the members of this association might find in this history some ground for pride in what they have thus far accomplished, is fully justified. Certainly, no other work of a kind similar to that upon which our efforts have been expended has recovered so wide a field, or has been a fundamentally and thoroughly done; and nowhere is there such an efficient organization as we now have for this kind of investigation.

[1] Journ. Am. Chem. 10, 402.

But, while we indulge in these pleasant reflections, let us not think of saying that our work is done, and that we shall rest on the laurels won. Happily no disposition to do this has been shown. Almost every year has witnessed some addition to the scope of our work. Last year the very important and difficult subject of the analysis of soils was taken up, a subject that needs very much such an organization as ours to make its methods practically useful, if they can be made so at all. I have faith that they can be made useful, at any rate in connection with field experiments with fertilizers.

New methods of estimating the constituents found in our agricultural materials and products are continually coming out. We should take into consideration such as are most promising, to learn whether they may not be useful to us. I would call attention especially to a method for phosphoric acid, heartily indorsed by some of the best iron chemists, in which, with the aid of a centrifugal machine, the amount of molybdate precipitate is determined in a very brief time by its volume. Certainly a method for phosphorus that will satisfy iron chemists is worth our consideration if we can gain anything by its use. Ulsche's new method for nitrates in fertilizers has been most heartily recommended to agricultural chemists in a very recent paper by such good authorities as Alberts and Hempel.

By thus appropriating from other departments of technical analysis whatever may help us, after subjecting it to our own rigorous system of testing, and holding fast to all that is good either in old methods or in new ones, being thus always both duly conservative and vigorously progressive, we shall serve our own purposes best, and we may reasonably hope perhaps to attain such a degree of importance that no more books on technical analysis will be published even in Germany entirely ignoring our work or our existence.

It would be ungrateful if in this connection I should not mention the assistance which has been afforded by the Department of Agriculture to this association, in making its history. It has given us a comfortable place for our meetings, provided us with all the needed facilities for recording our deliberations, and more than all for the publication of our annual reports. I am safe in saying, I think, that if out of our own pockets, or by begging others for means, we had been obliged to provide all that the Department has provided for us, we should have fallen far short of what we have actually accomplished. And to this should be added the recognition of the services in our behalf of that particular representative of the Department, our present secretary, who has been from the first one of our coworkers, and through whose instrumentality all this valuable assistance has come.

The PRESIDENT. The next thing in the order of business is the report of Prof. Rising, on fermented liquors. Prof. Rising, we all regret, can not be present, and his report will be read by the secretary.

Mr. Wiley, after reading a telegram from Prof. Rising expressing regret on account of his enforced absence this year, read that gentleman's report, as follows:

REPORT ON ANALYSIS OF FERMENTED LIQUORS.

By W. B. Rising.

As reporter on methods of "analysis of fermented liquors," I beg leave to submit the following report:

It was my privilege last year to visit a large number of wine chemists in Europe, and to have full and free discussions with them upon many points connected with wine analysis. The one wish, expressed by nearly every chemist consulted, was for a uniform method of wine analysis to be adopted by all who make wine analyses, so that their results can be compared. Especially is such uniformity of method wanted by a country exporting wine. The wine will be judged by the standard of the country into which it is imported, hence the necessity of uniform methods of examination. I think it not too much to say that the conclusions of the imperial "Health Bureau" (Gesundheits-Amt) are universally accepted, and that the deviations from them by individual chemists are fewer and fewer each year. In the list of determinations which are given below I have returned in many cases to the methods of the Berlin commission, for the sake of conforming to the world's methods.

In respect of specific gravity I prefer 15° C. to 25° C., adopted by the association two years ago, for the general reason that it is the world method, and recommend that 15° C. be adopted as the standard temperature for that determination.

In estimating extract I recommend that 50 cc. of wine (at 15° C.)—in case of sweet wines a less amount—be evaporated in a platinum dish 85 mm. in diameter, 20 mm. high, and of 75 cc. content, upon a water bath. The residue is heated two and one-half hours in a drying oven at 100° C.

(In sweet wines, *i. e.*, such containing more than .5 gr. of sugar in 100 cc. of wine, a lesser amount of wine is to be taken and diluted to 50 cc.; 1 gr. or at most 1.5 gr. of extract represents the maximum.)

The rest of Mr. Rising's report consisted in submitting without change the remaining parts of the methods of last year. These are printed under "Methods of Analysis."

The president stated that any other papers on this subject would come next in order. Discussion of the report was invited.

Mr. Crampton. This matter of the control of wines and alcoholic beverages has lately become an important matter, at least with some of us. I am the chemist of the Internal Revenue, and the subject has lately become one of official control. The act of Congress approved October 1, 1890, which some of you may be familiar with under the name of the McKinley bill, allows of the fortification of sweet wines by the addition of alcohol—grape brandy—which can be withdrawn free of tax within certain limits; that is, it allows certain wines, those containing at least 4 per cent of sugar, to be fortified with grape brandy to an extent not to exceed 24 per cent of alcohol in the finished product, and with an addition of not more than 14 per cent, so that this is now a matter of official control, and this makes it important for us to have methods which may be adopted by this association which we can utilize in our work. The methods as given by the association provide for the

complete analysis of wines and of alcoholic beverages, and are very good. Those which were adopted by the association last year were used in the Internal Revenue laboratory through the year in making analyses of wines under the provisions of the act; but we are obliged to provide in our office for the analysis of wines by rough methods. In order to ascertain whether a wine is competent to be fortified, it must be ascertained whether that wine conforms to the provisions of the act. Therefore we have adopted some rough methods for the estimation of alcohol and of sugar, which have been incorporated in the regulations of the U. S. Internal Revenue Office. I think it would be competent for this association to adopt these in addition to the methods recommended by the reporter, inasmuch as they are official methods, and it will doubtless add force to them to some extent to be adopted and placed among the methods used by this association. I have not a copy of the regulations with me, but I move that these rough methods for the estimation of sugar and alcohol in sweet wines be incorporated with the methods given by the reporter, if this is the proper place to make such a recommendation.

The PRESIDENT. It is moved that the rough methods mentioned by Mr. Crampton for determining the amount of sugar and alcohol in sweet wines be incorporated in the methods adopted by the association. Is that motion seconded, in order to bring it before the association?

The motion was seconded.

The PRESIDENT. It is now before the meeting for discussion. Are there any remarks?

Mr. PAYNE. The gentleman has just spoken of rough methods. I should like to know whether he means approximate methods.

Mr. CRAMPTON. Yes, sir. I would like to say that I would be glad to hear from any gentleman here on this subject of the analysis of alcoholic beverages. It is, of course, one not of so much general interest, as others, to the association.

The PRESIDENT. The chair would like to make one remark before proceeding to discussion. It is customary that all motions should be handed to the chair before they are stated to the meeting. I shall be obliged to ask that we go on in accordance with that common practice.

Mr. FREAR. I consider Mr. Crampton to be an exceedingly careful analyst, and have no doubt that the methods proposed by him are entirely adequate for the purpose, but I question the policy of adopting methods without knowing what they are, and I think the association should wait long enough to have them read before taking final action.

Mr. PAYNE. As a substitute for the motion before the association I move that the methods referred to be submitted to a committee, who should go over them and submit them to the association, in writing, for its action. Carried.

The PRESIDENT. I will name the committee later, and ask if there are any further remarks to be made on the report on the analysis of fermented liquors.

Mr. WILEY. I desire to call attention to the fact that Mr. Rising has referred in his report to a great many processes with which the members of the association are not familiar, and suggest that it would be well for the secretary to ask him to supply an outline of the methods referred to, not in common use, to be included in the report.

The PRESIDENT. It is important to have a careful description of these methods of analysis, and no objection being expressed, the secretary will make such request of Mr. Rising.[1]

Mr. LUPTON. I move that the report be accepted and adopted by the association.

At the suggestion of the president action was deferred until the committee could report on the other methods, when all could be adopted together.

Mr. Cooke presented the following report on dairy products:

REPORT ON DAIRY PRODUCTS.

By W. W. COOKE.

The coöperative work among the members of this association during the past years in the line of dairy products has been confined to butter and butter substitutes. For this reason it seemed best that the work this year should be on milk.

The first sample was sent out in February. It was a sample of whole milk that was brought to the station as soon as milked, cooled in ice water, thoroughly mixed, and put into pint glass fruit jars, the jars being filled to prevent churning of fat by agitation while on the road. The jars were sent by express the same day to all the experiment stations in New England and the Middle States, with a letter requesting them to analyze the sample for total solids, fat, and nitrogen, reporting methods used in the determinations.

Reports on the analysis of samples were received from the Maine Experiment Station, J. M. Bartlett; New Hampshire Experiment Station, F. W. Morse and C. L. Parsons; Massachusetts State Experiment Station, C. A. Goessmann; Connecticut State Experiment Station, E. H. Jenkins; Connecticut Experiment Station at Storrs, C. D. Woods; New York State Experiment Station, L. L. Van Slyke and W. H. Whalen; New Jersey Experiment Station, C. A. Cathcart; Vermont Experiment Station, J. L. Hills and B. O. White.

All of the chemists say that the sample arrived in good condition and was analyzed at once while it was still sweet. The following table gives the results as they were reported:

[1] In accordance with this action Mr. Rising was requested to furnish a description of the methods referred to in his report, but up to the time of going to press no response has been received.

[First sample: Whole milk, sweet.]

Chemist.	Specific gravity.	Total solids.	Fat.	Total nitrogenous matter.	Milk sugar.	Ash.
		Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
Bartlett, Maine	.....	[1] 12.99	[11] 3.88	[13] 3.31	[15] 5.80	
Morse, New Hampshire	1.0338	[2] 13.19	[8] 3.91	[12] 3.25	[15] 5.27	0.76
Parsons, New Hampshire	1.0338	[2] 13.12	[8] 4.03 [10] 3.90 [9] 4.02	[13] 3.12	[15] 5.23	0.79
Goessmann, Massachusetts	.....	[2] 12.91	[8] 3.81 [5] 3.83	[12] 3.37	[15] 5.72	
Jenkins, Connecticut	.....	[4] 13.02	[7] 3.91	[13] 3.44	[15] 5.67	
Woods, Connecticut	.....	[3] 12.97 [3] 12.95	[6] 3.99 [5] 3.86	[12] 3.47 [13] 3.47	[15] 5.56	
Cathcart, New Jersey	.....	[1] 12.96	[6] 4.04	[13] 3.50	[14] 5.70	[16] 0.72
Van Slyke and Whalen, New York	.....	[4] 13.17	[7] 3.80	[13] 3.63 [14] 3.69	[15] 5.71	
Hills, Vermont	1.034	[1] 13.06	[5] 3.97 [6] 4.07 [9] 4.02	[13] 3.49	[14] 5.71 [16] 4.85	0.84
White, Vermont	.....	[1] 13.08	[5] 3.99 [6] 4.07	[13] 3.63	[15] 5.42	
Average	1.0339	13.05	3.91	3.45	4.92	0.78

[1] Drying in air in boiling water oven.
[2] Drying with sand in air oven, 105° C.
[3] Drying with sand in boiling water oven.
[4] Drying with asbestus in boiling water oven.
[5] S. and S. fat-free paper.
[6] Ordinary paper, factored.
[7] Babcock asbestus method.
[8] Sand method.
[9] Beimling.
[10] Parsons.
[11] Babcock, volumetric.
[12] Kjeldahl, evaporating.
[13] Kjeldahl, not evaporating.
[14] Gunning.
[15] By difference.
[16] By polariscope.

METHODS.

The methods employed by the analysts were as follows:

Maine, Mr. Bartlett: Total solids by drying in air two and one-half hours at the temperature of boiling water; fat by the Babcock whirling method as described in Bulletin 24 of the Wisconsin Experiment Station; nitrogen by Kjeldahl.

New Hampshire, Mr. Morse: Total solids, on sand in a porcelain crucible, dried for two and one-half hours in the air bath at 105° C.; fat determined in this residue by transferring to a Mohr's extractor and treating for four hours with Squibbs ether, purified over calcium chloride, the extract dried for two hours in the air bath at 105° C.; nitrogen by Kjeldahl, evaporating to dryness before the addition of the acid; ash by evaporating over a low flame with the addition of nitric acid; specific gravity by Westphal balance.

New Hampshire, Mr. Parsons: Total solids same as Mr. Morse; fat the same, except that it was extracted for two hours; nitrogen by Kjeldahl without evaporation; ash the same but ignited in a muffle; specific gravity by the Westphal balance. Fat was also determined by the Beimling method as described in Bulletin 21 of the Vermont Experiment Station, and four days after, when the sample was sour, fat was determined in it by the Parsons method, as described in the first report of the New Hampshire Experiment Station.

Massachusetts, Mr. Goessmann: Total solids, evaporated on sand in porcelain dish and dried to constant weight in air bath at 100° to 105° C.; fat was determined by two

methods. In the first, the residue from the total solids was pulverized without removing from the dish, transferred to a filter, and exhausted with anhydrous alcohol-free ether. The fat obtained on the evaporation of the ether was dried in an air bath at 100° to 105° C. to a constant weight; in the second method the milk was put on S. & S. fat-free paper that had been extracted with alcohol and ether, treated in the Soxhlet extractor with more than 12 siphonings, dried in an air-bath at 100° to 105° C. to constant weight; nitrogen was determined by Kjeldahl, first evaporating the milk to dryness and using metallic mercury in place of mercuric oxide.

Connecticut, Mr. Jenkins: Total solids by drying on asbestus in a perforated tin tube to constant weight in a boiling-water oven; fat by extracting this residue with absolute ether for four hours, drying the fat in air in a boiling-water oven; nitrogen by Kjeldahl.

Connecticut, Mr. Woods: Total solids were made on sand in a platinum crucible, and also in a porcelain crucible, almost identical results being obtained by the two methods. Fat was determined by the Adams method on S. & S. fat-free paper, and also by the Adams method on ordinary filter paper No. 598, using the correction for the extractive matter in the paper, of 0.010 gr.; nitrogen by Kjeldahl, with and without evaporating the milk before the addition of the acid, the same results being obtained in both cases.

New York, Messrs. Van Slyke and Whalen: Total solids by the Babcock asbestus method, drying for two and one-half hours; fat, the residue extracted by ether for six hours; nitrogen by Kjeldahl and also by the Gunning methods, the results by the latter being a trifle higher than those by the Kjeldahl.

New Jersey, Mr. Cathcart: Total solids by evaporating in air in a boiling-water oven; fat by the Adams method, using ordinary paper, subtracting a constant factor for the extractive matter in the paper; nitrogen by Kjeldahl; ash by ignition with nitric acid.

Vermont, Messrs. Hills and White: Total solids by evaporating in air in a boiling-water oven; fat by the Adams method, using S. & S. fat-free paper, unextracted, and also the ordinary paper, No. 598, substracting a constant for the extractive matter in the paper, also by the Beimling method; nitrogen by Kjeldahl; ash by ignition with the addition of nitric acid; milk sugar by the polariscope.

DISCUSSION OF RESULTS.

Fat.—The only official methods for the past year have been the Adams method and the Babcock asbestus method. By reference to the methods used, it will be noticed that several others were employed in addition to those which are official. Judging from the results obtained, the sample of milk contained 3.91 per cent of fat. The average of the four determinations of fat by the official method, using S. & S. fat-free paper, is 3.91. The average of the two analyses by the Babcock asbestus method is 3.86. The average of the three analyses by the sand method is 3.92; and the average of the four determinations by what may be called the semichemical methods, those of Babcock, Beimling, and Parsons, is 3.93. The average of the four analyses, using the Adams unextracted paper and subtracting a constant, is 4.04.

It may be stated in general that the results are very good, and much better than one would be led to expect from the remarks that have been current among outsiders during the last few years in regard to the difficulty of making a correct analysis of fat in milk, and the great variations different laboratories obtained on the same sample.

Omitting the results obtained on unextracted paper, of which we will speak later, and the results obtained by the semichemical methods, the difference between the extremes is 0.24 per cent. All the results are within 0.12 of the average: three-fourths of them are within 0.09 of the average; the probable error is 0.06. This compares fairly well with the results obtained by members of this association in their chemical determinations. The limit of error is within three one-hundredths of the total

amount of fat present, and the probable error is twelve thousandths of the total amount present.

The results obtained by those analysts who used unextracted paper and deducted a constant are worthy of notice. It will be seen that higher results are obtained by this method than by the others. A special study was made of this phase of the subject to determine the cause of these results, and it was found that after milk had been put on paper the paper yields up more of its substance to absolute ether than it did at first. This is true of sweet milk, and to a much greater degree after the milk becomes sour, and this amount, though constant for one sample of milk and one kind of paper, is quite variable between different samples of milk of different ages, and with different papers, so that it becomes impossible to make a factor which can be taken as a constant in all cases. As the process of extracting the paper is quite slow and tedious, and the paper already extracted is quite expensive, it seems very desirable that some way should be found by which unextracted paper can be used and still correct results be obtained. After a good deal of investigation and a large number of trials we have found that the addition of a few grains of solid sodium carbonate to the milk prevents it from having this action on the paper, and allows a constant to be obtained and used. The small quantity of sodium carbonate necessary for this does not perceptibly increase the quantity of milk, and no correction need, therefore, be made for it.

Total solids.—The work in total solids is eminently satisfactory. The official methods allow sand to be used or not, and both methods have been employed. The average of all the results gives 13.05 per cent total solids. The average of the four determinations without sand at a temperature of boiling water is 13.02, and of the three determinations with sand at the same temperature 13.04; of the two determinations with sand at 105° C. in an air bath is 13.10. It would seem that evaporating in a bath without sand is fully as accurate as with it, and if the residue is not to be used for a further determination of fat, it is the preferable method. The difference between the extreme results on total solids is 0.38. All are within 0.15 of the average, and three-fourths are within 0.10 of the average. The probable error is 0.08 per cent, or only six-thousandths of the total amount present.

Total nitrogenous matter.—This was determined in all cases by Kjeldahl, and with only two important differences. The official method makes no statement as to whether the milk shall or shall not be evaporated to dryness before adding the acid. Some of the chemists evaporated and some did not. Omitting one low result, the average of all is 3.45 per cent, which is the nitrogen multiplied by 6.25. Seven chemists did not evaporate before the addition of acid, and obtained an average of 3.36, making it evident that evaporation tends to produce low results, although Mr. Woods obtained identical results (both of them very close to the average) by the use of both methods. The difference between the extremes of the determinations is 0.51; all are within 0.20 of the average, with a probable error of 0.10. Although this work does not show such a close correspondence as the fat, yet, considering the fact that it has almost no commercial value, and those who made the determinations are not in the habit of making these analyses so often as they are the determinations of total solids and fat, the results are on the whole satisfactory.

Ash.—This was in all cases determined by the official method, adding nitric acid to the milk and evaporating to dryness. But four chemists made the determination, and their results agree closely, giving 0.78 as the average.

Milk sugar.—This is usually determined by difference. In the present case most of the chemists determined neither ash nor milk sugar, so that the amount found by subtracting from total solids the fat and the nitrogenous matter, gives the sum of the ash and the milk sugar. If from this were subtracted the average 0.78 for the ash, it would give the milk sugar by difference. The results can hardly be said to be satisfactory, unless we remember that in this determination by difference are accumulated and magnified the errors in the other determinations. The greatest difference is 0.61,

or twelve one-hundredths of the total milk sugar present. All the determinations are within 0.35 of the average, with a probable error of 0.16, or three one-hundredths of the total amount present. The average sum of the milk sugar and ash is 5.70, which, if we subtract the average ash, 0.78, leaves the milk sugar by difference, or 4.92. The only direct determination we made with the polariscope was 4.85, a fairly close approximation.

Second sample, sour milk.—The results on this first sample were so satisfactory that it was deemed advisable to test the various chemists on a sample of sour milk that was in a very poor mechanical condition. For that purpose a lot of sweet cream was churned, and the resulting buttermilk divided, part kept cold, and part allowed to become warm and sour. It was intended by this means to obtain the results of the chemists on the same sample, both in a good and in a poor condition. Through a series of unfortunate circumstances and accidents the samples were delayed in transit, and both were thoroughly sour when they arrived, so that the results on the two samples become duplicates. The results are given in the following table:

Report on dairy products.

[Second sample: Sour buttermilk.]

Chemist.		Total solids.	Fat.	Method used in determining fat.
		Pr.cent.	*Pr. ct.*	
The sample when sweet contained		9.98	0.69	Adams S. and S., fat-free.
New Hampshire, C. L. Parsons	I	9.74	0.50	Ammonia added, dried on sand and extracted.
	II	9.66	0.54	
Massachusetts, C. A. Goessmann	I	7.68	0.39	Dried with sand, and was held with ether on a filter.
	II	9.07	0.24	
Connecticut, C. D. Woods	I	9.41	0.65	Ammonia added, S. and S., fat-free, paper.
			0.64	Ammonia added, common paper, corrected.
	II	9.40	0.64	Ammonia added, S. and S., fat-free, paper.
			0.63	Ammonia added, common paper, corrected.
New York (Cornell), H. Snyder	I	9.54	0.40	Babcock whirling method.
	II	9.61	0.40	Do.
New York (Geneva), L. L. Van Slyke	I	9.67	0.40	Do.
	II	9.61	0.20	Do.
Vermont, sour sample	I	9.97	0.65	Adams S. and S., fat-free, with ammonia and soda.
	II	9.74	0.64	

The sample when sweet, as determined at the Vermont Station, contained 9.98 per cent total solids and 0.69 per cent of fat; total solids being determined by evaporating in air at the temperature of boiling water, and the fat by the Adams method, using S. & S. fat-free paper.

At the same time that these samples were sent, an extra set was sent to the express office in New York City, with the request that it be returned to the station. It was received and analyzed at presumably the same time that the other samples were being analyzed at the different stations. The results are given in the table as the Vermont sour samples. The methods used by the other chemists for analysis of these samples were the same as those employed on the earlier sample, except as noted above in the table.

DISCUSSION OF RESULTS.

Fat.—The determination of fat was, of course, the most important determination, and it was on account of this that the samples were sent out. The table will show two distinct sets of results, one where ammonia was added to the sample before analysis, and the other where it was not added. The results obtained by Mr. Woods by the Adams method, after adding ammonia to the sample, are especially worthy of notice.

In this case he added 25 cc. of strong ammonia to 500 cc. of milk, and obtained results practically the same as those obtained on the sweet milk. Mr. Parsons used only enough ammonia to neutralize the acid present, and his results are somewhat lower, while the rest of the analysts obtained scarcely half the amount of fat actually present in the sample. At the Vermont Station ammonia was tried, and also carbonate of soda, the results obtained agreeing very closely with those reported by Mr. Woods; that is, the results are almost as high as they were on the fresh sample.

Total solids.—As might be expected, these are lower on the sour samples than on the sweet, and so far as reported no station attempted to make any calculation to determine what the total solids were in the milk when it was sweet. In general, the total solids are 9.60 per cent, or a drop of 0.40 from the original sweet milk. It is probably possible to use some method of analysis that will enable the original per cent to be quite accurately determined, and it is a subject which it would be well for this association to investigate.

SEMICHEMICAL METHODS.

By vote of the association, the reporter was requested to examine the new quick methods of fat analysis. The only ones now on the market that need to be considered are the Babcock and the Beimling. Each gives accurate results on whole milk in good condition, when all the precautions as given by the authors are followed. If carelessly handled, the tendency of the Babcock is to give low results, and of the Beimling to give high. In our own laboratory we use the Beimling method in the Babcock bottles and machine. On sour samples, on skim milk, and on buttermilk neither method gives us as accurate results as the gravimetric, though both are accurate enough for commercial work, and for a large part of the station experimental work. Our station has made over 7,000 determinations of fat in the last year, and most of them have been by one or the other of these methods. The two methods have been tried side by side on several hundred samples, and the average results are identical.

METHODS RECOMMENDED FOR MILK ANALYSIS.

Water.—Same as in Bulletin 28.

Total nitrogenous matter.—Place in a Kjeldahl digestion flask a known weight (about 5 grs.) of milk, and proceed, without evaporating, exactly as described for this method by the nitrogen reporter.

Fat.—Method of Adams, modified by Wiley.

Coils made of thick filter paper, cut into strips 2.5 by 25 inches, are thoroughly extracted with ether and alcohol, or the weight of the extract corrected by a constant obtained for the paper. If this latter method is used, a small amount of anhydrous Na_2Co_3 should be added to the milk. The coil, dry end down, on a piece of glass, is dried at the temperature of boiling water, transferred to siphon extraction apparatus, and extracted by at least 12 siphonings with absolute ether, or petroleum spirit boiling at about 45° C. The extracted fat is dried in hydrogen and weighed.

If the milk is sour, add about 10 per cent by weight of strong ammonia water and a small quantity of anhydrous Na_2Co_3, correcting the results for the ammonia added.

Alternative method: Same as in Bulletin 28.

Sugar.—Same as in Bulletin 28.

Ash.—In a weighed platinum dish put 20 cc. of milk from a weighing bottle, add 6 cc. HNO_3, evaporate to dryness, and burn at low red heat until ash is free from carbon.

At the conclusion of Mr. Cooke's report the president announced that the committee to consider the additional methods for the determination of alcohol and sugar in wines would consist of Messrs. Crampton, Payne, and Frear.

Mr. FREAR. I would like to ask whether metallic mercury is used in the Kjeldahl determination. My assistants complain that where metallic mercury is used instead of mercuric oxide, they are troubled a great deal with frothing.

Mr. COOKE. Prof. Goessmann said in his report that he used metallic mercury, but did not say how he liked it.

Mr. Cooke presented the following paper on—

ANALYSIS OF SOUR MILK.

By W. W. COOKE.

This association has never taken any official action in regard to the analysis of milk in any other condition than sweet. The great extension in milk testing in the last few months, and especially the testing of buttermilk at creameries, rendered it advisable that this association should take some official action in regard to the best methods in handling sour, lobbered samples of either whole milk, skim milk, or buttermilk. That the present condition of the analyzing of such samples is not satifactory is abundantly shown by the figures already presented on the analysis of the sample of sour milk.

In order to better understand the problem before us it may be well to present in a few words the changes that take place during the souring of milk. Milk sugar is the part that changes most in the souring. This absorbs one molecule of water and breaks up into lactic acid, and this lactic acid on further souring breaks up into butyric acid, carbonic acid, and free hydrogen. The formulas are as follows:

Milk sugar. Lactic acid.

$$C_{12}H_{22}O_{11} + H_2O = 4\ C_3H_6O_3$$

Lactic acid. Butyric acid.

$$2\ C_3H_6O_3 = C_4H_8O_2 + 2\ CO_2 + 2\ H_2$$

Statements are made by various authors that lactic acid is partially volatile at 100° C.; that butyric acid is completely volatile at that temperature; also that ammonium lactate decomposes at 100° C., the ammonia being of course dispelled. The statements are also made that the lactic acid and butyric acid are completely soluble in ether; that the alkaline salts of each acid are entirely insoluble in ether; that sodium carbonate is insoluble in ether; that sodium hydrate is soluble.

In order to test the truth of these statements, and also to determine the behavior of milk under different conditions, the following experiments were made. Most of the analytical work was performed by Miss Ellen P. Cooke:

Half normal solutions were made of pure lactic acid and pure butyric acid; 5 cc. $\frac{N}{2}$ lactic acid should contain 0.225 grs. lactic acid; 5 cc. $\frac{N}{2}$ butyric acid should contain 0.220 gr. butyric acid; 5 cc. $\frac{N}{2}$ lactic acid, heated for several hours in various-shaped dishes at 100° C., gave as a residue 0.163, 0.144, 0.102, 0.152 gr., and in no case was a constant weight obtained. The amount of loss seems to be governed by the shape of the dish and the time of heating. The first quarter of the weight was driven off rapidly, and after that the loss in weight was slow, never amounting to more than one-half the lactic acid. When an excess of ammonia was added to 5 cc. $\frac{N}{2}$ lactic acid and heated for several hours at 100° C., there was a slight loss in weight, but nothing to indicate that there was a complete disintegration of the ammonium lactate. The theoretical weight of ammonium lactate would be 0.277, and 0.256, 0.246, 0.248, 0,266 were obtained, or about 90 per cent of theory. Half-normal butyric acid, both alone and after neutralizing with ammonia, we find to be completely volatile at the boiling temperature of water.

The next experiment tried was the adding of lactic acid and butyric acid to sweet skim milk, and then evaporating the mixture at the boiling temperature of water in an open, flat nickel dish. The lactic acid alone increased the total solids up to 80 per cent of its own weight; that is, one-fifth of the lactic acid seems to be volatile under these conditions. When more than sufficient ammonia was added to neutralize the lactic acid the total solids were increased up to 80 per cent of the theoretical amount of ammonium lactate. Butyric acid alone added to the milk left 10 per cent of its own weight in drying; butyric acid and an excess of ammonia left 45 per cent of the theoretical weight of the ammonium butyrate. These results would seem to show that lactic acid and ammonium lactate are but little volatile when present in milk and dried down in the mass; that butyric acid alone is mostly volatile, and that about half of ammonium butyrate comes off with an ordinary amount of drying.

A known amount of $\frac{N}{2}$ lactic acid was put on to strips of S. and S. fat-free paper as prepared for milk analysis, dried for several hours at 100° C., and then soaked in water, and the resulting liquid titrated to ascertain how much of the lactic acid remained. It took less than 1 cc. of $\frac{N}{2}$ alkali to neutralize the solution, showing that four-fifths of the lactic acid either evaporated or had been changed to such a state that it was not soluble in water. When the same was tried with butyric acid it all evaporated. When lactic acid was put on the paper, and then an excess of ammonia added to the paper, dried, and afterward soaked in water, 80 per cent of the lactic acid was recovered in the solution, the results in these cases agreeing exactly with those obtained when the lactate of ammonia was evaporated in the milk, whereas the results on lactic acid alone were just the opposite, only one-fifth evaporating from the milk, while four-fifths evaporated from the paper.

The same amount of lactic acid, 5 cc., was put upon the same papers, dried, and then extracted for three hours with absolute ether in a Soxhlet extractor, the paper alone giving an extract of 0.001 gr., and the lactic acid on the paper giving an extract equivalent to less than one-twelfth of the total lactic acid added to the paper. Lactic acid and an excess of ammonia gave an extract equivalent to one-twentieth of the theoretical amount of ammonium lactate. Lactic acid neutralized with sodium carbonate gave an ether extract of about 3 milligrams, and the same was obtained when the lactic acid was neutralized with ammonia and carbonate of soda. After the papers that had received the pure lactic acid had been extracted with ether for three hours they were soaked in water for three hours, and the aqueous extract on titration showed the presence of 37 to 76 milligrams of lactic acid. Thus in some unaccountable way lactic acid, shown by the water to be on the paper, was not extracted by the ether. Butyric acid gave no ether extract, whether added alone to the paper, or neutralized with ammonia.

So far it seems probable that ammonia alone added to sour milk would prevent all but a very small ether extract of the lactic acid, while carbonate of soda would prevent most if not all of this small amount. It also seems probable that if lactic acid formed in the milk it would not be dispelled by drying at 100° C., and some of this lactic acid would be dissolved by the ether, and thus give higher results on the sour milk than on the sweet. Such, however, does not seem to be the case in actual practice, and I am unable as yet to account for the results. As a fact, the ether extract of the sour milk is always lower than that obtained from the same milk when sweet. A sample of skim milk which gave 0.575 per cent of fat when sweet, as tested by the Adams method on S. and S. fat-free paper, gave two days after, when slightly sour, 0.45 per cent of ether extract, and when five days old and wheyed off, 0.37 per cent ether extract. When the sample was three days old the addition of ammonia in slight excess brought back the ether extract to 0.56, or the same as when sweet. The addition of the same amount of ammonia, and also a little carbonate of soda, gave the same results. When the sample was five days old the same treatment—that is, the addition of ammonia in slight excess, and carbonate of soda—gave an ether extract of

0.33, which is practically the same as was obtained without adding anything to neutralize the acid.

Another sample gave very much the same results—when sweet, 0.72 per cent of fat; when wheyed off and neutralized, 0.54; neutralized with slight excess of ammonia, 0.46; neutralized with carbonate of soda, 0.56.

In no case did the results on the sour milk equal those obtained on the sweet milk; the samples gave with ammonia only three-fourths of the fat per cent, and neutralized with carbonate of soda only one-half.

A test was also made to determine whether the carbonate of soda when added to sweet milk would make any difference in the ether extract. A sample gave 2.44 per cent of fat when sweet, and when carbonate of soda was added to the sweet milk an ether extract of 2.40.

A further investigation led to the conclusion that the addition of a considerable quantity of ammonia to sour milk would break up the curds completely, but even this alone was found in some cases not to bring back the fat to its original quantity. When, however, carbonate of soda was also added the results were eminently satisfactory.

	Sweet.	Sour, plus NH_3 and Na_2CO_3.
	Per cent.	*Per cent.*
Skim milk	0.48	0.48
Whole milk	2.44	2.34
Skim milk	1.25	1.32
Buttermilk	0.96	0.92
Average	1.03	1.01

It would seem then that the trouble with sour milk is principally mechanical, the particles of fat being entangled in the clotted casein, so that the ether will not extract them, and that in some unaccountable way the lactic acid, after the thorough drying on the paper, either evaporates or is no longer soluble in ether. The addition of 10–12 per cent of ammonia by weight reduces the milk again to a state of fluidity, and the addition of a small quantity of carbonate of soda insures the alkalinity of the mass after the ammonia has evaporated.

During the course of this investigation some interesting results were obtained on the use of ordinary unextracted filter paper for the Adams method. The paper used was No. 598, and a very even and good sample. It was moistened, cut into strips of the same size, and the extraction of several of these strips for several hours with boiling ether gave a very uniform extract of 0.0067 gr. Sour skim milk, which by the S. and S. fat-free paper gave 0.08 per cent fat, gave on this ordinary unextracted paper, after taking out this constant of 0.0067 gr., a residue of 0.53 per cent ether extract. When this sample was neutralized by ammonia there was still obtained an ether extract of 0.31 per cent, but upon neutralizing with carbonate of soda and subtracting the constant the residue was the same as obtained on the S. and S. fat-free paper. The following table gives the results obtained on this and four other samples of milk by the same method:

	S. S.	Ord.	Na_2CO_3.
	Per cent.	*Per cent.*	*Per cent.*
1 Sour	0.08	0.53	0.08
2 Sour	0.19	0.57	0.13
3. Sweet	2.44	2.97	2.41
4. Sweet	3.46	3.73	3.45
5. Sweet	3 37	3.57	3.31

These figures show quite conclusively that, if the milk is first neutralized with carbonate of soda, it is possible to use the ordinary cheap unextracted paper for both sweet milk and sour milk, taking out the constant determined for the paper. The probable explanation seems to be that alkaline milk has no action on the paper, and the nonvolatile soda keeps the solution alkaline through the whole process.

Total solids.—The problem of the determination of solids in sour milk has engaged the attention of the English analysts for some time, and there is a wide diversity of opinion among them as to the best method of analyzing, or how much loss there is in the total solids of milk when it is sour. That there is a loss is known to everyone who has made total solids in sweet milk, and again on the same sample when sour. This drop varies all the way from .20 to nearly 1 per cent.

Theoretically, if the lactic acid is not volatile, there should be no drop in the total solids until the lactic acid begins to break up into butyric acid, and this does not happen until after the milk has thoroughly lobbered and is beginning to whey off. We find, however, in practice, that the total solids begin to drop the moment that acidity is distinguishable by the test paper, and drop more and more the longer the milk stands.

The most promising method for determining total solids in sour milk seems to be the addition of some nonvolatile alkali to neutralize the lactic acid and prevent its evaporating. It is necessary, however, to add a known quantity of the alkali of known strength, in order to make a correction for the amount of solids added in the alkali.

The best alkali to use is decinormal sodium hydrate. When this is added to milk containing lactic acid, the soda unites with it to form sodium lactate and water. Each cubic centimeter of $\frac{N}{10}$ NaHO adds 0.0022 gr. to the weight of total solids.

The milk is thoroughly mixed, and 10 cc. put into a beaker with 50 to 100 cc. water, and titrated with $\frac{N}{10}$ alkali, using phenol phthalein as an indicator. Suppose 8 cc. is required to give the pink tinge, then if 10 cc. require 8 cc. to neutralize, 5 cc. of milk will require 4 cc. of alkali.

The 5 cc. of milk is put in a tared flat-bottomed dish, such as is usually used for total solids, 5 cc. of water added, and the 4 cc. of alkali. The water is added to make the alkali unite more evenly and perfectly with the milk. From the final weight $4 \times 0.0022 = 0.0088$ gr. is subtracted for the alkali, leaving the total solids in the milk. This method gives excellent results when milk has soured and is lobbered, but is not wheyed off. The following are some of the results that have been obtained:

	Sweet.	Lobbered.
Buttermilk ..... per cent..	1.88	8.82
Skim milk ..... do.....	10.84	10.78
Buttermilk ..... do.....	9.98	9.96
Skim milk ..... do.....	10.38	10.25
	10.93	10.85

When, however, the milk has wheyed off, this method will not work, for in the formation of butyric acid there has been an actual loss of substance from the milk in the shape of carbonic acid and free hydrogen.

The following method has given us fairly good results the few times it has been tried on samples six to eight days old and thoroughly wheyed off:

EXAMPLE.

4.931 gr. sour milk + 5 cc. water + 5.50 cc. $\frac{N}{10}$ NaHO = 0.521 gr. solids.

5.50×0.0022 gr. = 0.012 gr. solids in the alkali.

$0.521 - 0.012 = 0.509 \div 4.931 = 10.32$ per cent solids.

10 gr., diluted and titrated required 10.90 cc. $\frac{N}{10}$ soda.

10 gr., diluted, boiled, and titrated, required 6.40.

$10.90 - 6.40 = 4.50$ cc. $\frac{N}{10}$ butyric acid driven off.

$4.50 \times 0.09 = 0.41$ per cent $CO_2 + H_2$ lost in the milk.

$10.32 + 0.41 = 10.73$ per cent solids in original milk.

This milk when sweet had contained 10.84 per cent total solids.

Another sample gave 8.81 which sweet had analyzed 8.85.

To a known weight of the milk (about 5 grams) in a tared flat dish, add 5 cc. of water and enough decinormal sodium hydrate to just neutralize the acid present, this amount having been previously determined in a separate portion diluted with water in a beaker, using phenol phthalein as an indicator. Dry and weigh as usual. From the amount of solids thus obtained subtract 0.0022 gr. for each cc. of decinormal soda used: the remainder is the total milk solids.

Fat.—Add about 10 per cent by weight, of strong ammonia water, and a small quantity, not more than 0.1 grain, of anhydrous sodium carbonate, correcting the results for the ammonia used. The analysis can be made by any of the official methods for the determination of fat.

Make total solids as above. To 10 gr. of milk in a beaker add water and boil briskly for half an hour, replacing occasionally the water that evaporates, and titrate. Add 0.09 per cent to the total solids for each cc. less of $\frac{N}{2}$ soda required to neutralize the 10 gr. after boiling, than required before boiling.

Mr. Patrick presented the following paper:

COMPOSITE MILK SAMPLES IN THE LABORATORY.

By G. E. Patrick.

Composite milk samples for use at creameries as a means of saving labor in the valuation of milk by any of the "oil tests," I first proposed (in detail) in Bulletin No. 9 of the Iowa Experiment Station, May, 1890. The preserving agent there recommended for preserving the samples was corrosive sublimate, $HgCl_2$, numerous experiments having shown that it preserves the *mechanical* as well as the chemical condition of milk better than any of the other common antiseptics. For use in creameries, I insisted that the sublimate have mixed with it some suitable aniline color, as a guard against accidental poisoning, and, to hasten solution in the milk, admixture of common salt, NaCl, was recommended.

For six months past I have employed the same principle in the laboratory, in analyzing the milk of experimental cows, not only for fat (by one of the "oil tests"), but also for solids, gravimetrically. (See Iowa Station Bulletin No. 13, page 29, May, 1891.)

For this purpose the preservative is of course used without admixture of aniline color or common salt, as these would bring error in the results on solids.

The corrosive sublimate is powdered finely and passed through a very fine gauze sieve. Only a very small amount is needed to preserve milk samples five or six days without material change; and five days is as long as such keeping is desirable in most experiments on milk production. For keeping five days, 0.125 gr. of the $HgCl_2$ is sufficient in cool weather, and 0.200 gr. in summer weather, provided the daily samples are 50 cc. each, making the complete composite sample 250 cc. The theoretical error thus introduced in the result on solids is only 0.05 per cent with the smaller amount and 0.08 per cent with the larger; the former figure is within the "limits of error" in ordinary routine work, and the latter nearly so if not quite. Many comparative trials have, however, convinced me that there is a *very slight* loss in the solids of milk preserved for five or six days, but that it rarely exceeds 0.05 per cent; therefore it is my custom to neglect correction for the $HgCl_2$ when it amounts

to only 0.05 per cent, and when it amounts to 0.08 per cent to correct by deducting 0.03 per cent. These corrections are accurate enough for use in routine work, by the method of drying in air on fine asbestos in open watch glasses, the method which I have thus far employed; doubtless finer work could have been done, and perhaps more accurate corrections found by the method of drying in hydrogen, had time permitted the employment of this method.

The following test determinations were made by Mr. E. N. Eaton, assistant chemist in this station:

I. Experiments in which the entire sample of milk was preserved for the time named, no daily additions of fresh milk having been made.

(*a*) With 0.05 per cent of $HgCl_2$ added; no correction is made for this in final results.

Sample.	Solids in fresh milk.	No. of days preserved.	Solids in preserved milk.
	Per cent.		*Per cent.*
No. 1	10.34	5	10.34
No. 2	12.35	5	12.45
No. 3	10.95	5	10.92
No. 4	11.13	5	11.13

(*b*) With 0.10 per cent of $HgCl_2$ added; results corrected by deducting 0.05 per cent:

Sample.	Fresh.	No. of days.	Preserved.
	Per cent.		*Per cent.*
No. 5	11.27	8	11.27 − 0.05 = 11.22
	11.27	9	11.33 − 0.05 = 11.28

(*c*) With 0.65 per cent $HgCl_2$ added by mistake; results corrected by deducting 60 per cent.

Sample.	Fresh.	No. of days.	Preserved.
	Per cent.		*Per cent.*
No. 6	13.47	8	13.94 − .60 = 13.34
	13.47	9	14.06 − .60 = 13.46

This last trial (*c*) indicates that the usual amount of $HgCl_2$, viz, .05 per cent, is as efficient as a much larger quantity.

II. Composite samples; fresh milk added each day; $HgCl_2$ added = .05 per cent on entire composite sample. Results not corrected.

Cow.	Mean of results on daily samples analyzed separately.	No. of days.	On composite samples.
	Per cent.		*Per cent.*
No. 114	10.53	5	10.45
No. 115	10.80	5	10.78
No. 37	14.30	5	14.36
No. 38	14.60	5	14.58

In warm weather I prefer the use of .08 per cent or .10 per cent of $HgCl_2$, with a correction of .03 or .05 per cent.

Lightning or Mason jars are convenient receptacles for the composite samples. The mercuric chloride is weighed out and placed in the jar at the time of adding the first daily sample or before. Upon the addition of each daily sample to the composite, the latter should be well mixed by a rotary motion—not by shaking—in order to redistribute the cream throughout the whole; and this mixing should be done every day, whether the samples be added every day or not.

Sometimes, especially in warm weather, the composite sample will have floating upon its surface small flecks of milk-solids; these can be broken up, and the sample brought into almost perfect mechanical condition, by means of a stiff test-tube brush used as a pestle inside the jar, rubbing the flecks to pieces against the walls of the latter.

One must guard against error from the rising of minute flecks of milk-solids to the surface while weighing out the charge; this is easily done by inverting the weighing pipette once or twice just before running out the charge upon the asbestos.

It hardly needs saying that in summer the composite samples should he kept in as cool a place as possible; ice or cold water would of course be useful.

EXPERIMENT STATION, AMES, IOWA, *August* 10, 1891.

Mr. VAN SLYKE. I move that the report on dairy products be taken up and each division of the subject considered separately. Carried.

Mr. COOKE. The analysis of butter has not been touched by my work during the year, and while that subject would, of course, be considered by the association, as others have undoubtedly worked with butter, I suggest that, the subject being fresh in mind from the reading of the report, it would be well to take up the methods for the analysis of milk, and consider these first.

The PRESIDENT. There is no objection to that course, and, to begin, it is proper that a motion be made in regard to the determination of the water by the official method.

Mr. WOODS. In the case of sour samples is not the addition of ammonia, in order to get a fair sample, as important in determining the water and other constituents as in the analysis of the fat?

Mr. COOKE. It is not necessary. It is possible to get from a very bad sample 5 cubic centimeters or 5 grams, which would correctly represent the average character of the mass. The work of various members of the association shows that to be possible, and, if soda be used for titrating, ammonia could not, of course, be used.

Mr. FARRINGTON. It is essentially necessary to add some alkali before taking the sample for analysis, because, if a sample be agitated while in a sour condition, the cream would be churned and would separate. Experience has shown that it is almost always necessary to neutralize the acid in a sour sample, in order to get it in proper chemical condition for weighing a small portion.

Mr. COOKE. In regard to the point referred to by Mr. Farrington, I will say that if the temperature be reduced with cold water to 45°, or, better still, 40°, the sample can be mixed as much as desired without danger of churning.

After some discussion as to the order of procedure in the consideration of the official methods, Mr. Woods moved that the topics be considered one by one, and then the method be adopted as finally settled by discussion. Carried.

Mr. LUPTON. I move that the method for determining the water in milk analysis be continued without alteration for the ensuing year.

The motion was adopted.

Mr. COOKE read again his second recommendation, and, on motion, it was adopted.

The third recommendation, in regard to the determination of fat, was read.

Mr. WOODS. I move the adoption of the recommendation, with the omission, however, of the last sentence, which I think is unnecessary, unless the title of the method were changed so as to include sour milk. If any methods for sour milk analysis are adopted, they should be separate from those for milk.

The PRESIDENT. Can not Mr. Cooke specify more carefully the small amount of anhydrous sodium carbonate?

Mr. COOKE. It is difficult to specify the exact amount, but a tenth of a gram will be an abundance for a pint of milk.

Mr. JORDAN. Will an amount of sodium carbonate, or any other alkaline compound added in excess of the actual acid present, not act upon the fat and saponify it?

Mr. COOKE. I took sweet milk and made a determination of the fat, and then added sodium carbonate and made the determination again, with practically the same results. I suppose that some saponification occurred, but it was so exceedingly small that very close analytical work failed to show it.

Mr. BABCOCK. I have made several experiments similar to that mentioned by Mr. Cooke, and found no indication that any saponification took place, even when a considerable excess of carbonate of soda was used.

Mr. WOODS. There does not seem to be much danger in adding the amount mentioned by Mr. Cooke, because that would be at the ratio of about a milligram to every 5 grams of milk, which would be exceedingly minute.

Mr. BABCOCK. Any one who had attempted to saponify butter fat, even with caustic alkali and heating for hours in the strength of solution referred to, would recognize the fact that such fats could not be easily saponified. I do not believe there is any danger of saponification in using an alkaline carbonate, as in this case, even if a large excess were added.

The motion of Mr. Woods was then put and carried, and the president stated that the suggestion of the reporter in regard to sour milk would be passed by for the present, to be considered later.

On motion, the alternate method for estimating water and fat in milk was adopted without alteration.

Mr. VAN SLYKE. I move that the Babcock centrifugal and Beimling methods be adopted as alternate methods for the determination of fat in milk.

Mr. WOODS. I do not favor the adoption of semichemical methods. The Beimling method is a patented method, and it is unwise for the association to adopt patented methods as official methods.

Mr. COOKE. The Beimling method is claimed to have been patented, but the patents are worthless, the Beimling method being almost without exception used, at present, in the Babcock bottles, which process is just as free as any chemical method. Besides, the Beimling Company has broken up, and there is no one to sue on the patent if it were good for anything.

Mr. VAN SLYKE. In regard to the adoption of semichemical methods, I ask for information in respect of how much chemical action there is in dissolving butter fat or milk fat by ether; whether that is any more chemical than separation by whirling.

Mr. LUPTON. I believe that a committee was appointed last year to report upon these different methods, such as the Babcock and other methods.

Mr. VAN SLYKE. I think no formal committee was appointed. The secretary was requested to insert in the report a description of the methods.

Mr. JENKINS. I am delighted with the Babcock method as used in our laboratory, and the results obtained by its use agree very closely with those obtained by the gravimetric method. We use it almost altogether, and yet I hesitate about agreeing to its adoption as one of the recommended methods of the association, because, in an accurate scientific investigation all the apparatus used would have to be tested, there being a possibility that the bottles might not have been accurately tested by the apparatus-makers.

Mr. Van Slyke's motion was then put and lost.

Mr. COOKE. I move that the method for the estimation of sugar be the same as before, but desire to call attention, in this connection, to the alumina cream for precipitating the impurities, which had been brought into notice in the method of sugar analysis for the Internal Revenue Department. I have not had time to apply this to the analysis of milk, but its use in work with sugar has given satisfaction, and it might on further investigation prove to be what is wanted in the determination of milk sugars.

Mr. Woods seconded Mr. Cooke's motion, and it was carried.

Mr. VAN SLYKE. I move that the alternate method for the estimation of sugar in milk be adopted without change. Carried.

Mr. Cooke read his recommendation in regard to the estimation of ash.

Mr. VAN SLYKE. Why should so large an amount as 20 cubic centimeters of milk be used? We have made a good many parallel

determinations, using from 2 to 5 cubic centimeters, and obtained results which agree perfectly. It requires a good deal of time to evaporate so large an amount as 20 cubic centimeters. I do not feel like making a motion to change the method without having some more special work done on it, and in asking the question my thought is to bring it before the association, in order to have the reporter for the ensuing year pay special attention to this point.

Mr. WOODS. I desire to call attention to the action of the association last year in inserting the word *platinum* in reference to the dish that is to be used in the determination of water. I can see no reason why a porcelain dish might not be used just as well for determining the ash as the water, and I move to amend the method by striking out the word *platinum*. Carried.

Mr. BARTLETT. Why should nitric acid be used in determining the ash? I have never used it in my work.

Mr. VAN SLYKE. Oxidation takes place much more rapidly, as experiments with duplicate samples show.

Mr. Cooke's motion, as amended, was then put and carried.

The hour being 12:30 p. m., a recess was taken.

AFTERNOON SESSION, THURSDAY.

The convention was called to order by the president at 2 p. m.

Mr. COOKE. I have no report or recommendation to make in regard to the analysis of butter.

Mr. BABCOCK. I move that the methods for the examination of butter described in the last report be adopted for the ensuing year. Carried.

Mr. PENNY. I move that the method of saponification by means of soda dissolved in glycerine, recently proposed by Drs. Leffman and Blake, of Philadelphia, be made a subject of investigation for the coming year.

As an excuse for proposing the new method, I will say that it differs from those in use at present in using glycerine as a solvent in the place of alcohol, and, as any one can see who is familiar with the methods for determining volatile acids, all loss of time would be avoided, and where the old method consumed two, three, or four hours this method will attain the same results in forty minutes, including thirty minutes for distillation. The saponification need not last more than five minutes.

Attention should be invited to these three points of inquiry: First and chiefly, does the glycerine-soda mixture give identical results with those of the present method? Secondly, if the free fatty acids are not dissolved before distillation, are the results the same? Thirdly, does a block-tin condenser give the same results as a glass condenser? From my own experience I think an affirmative answer will be found in each

case. I am satisfied that there would be no difference in results between the method in use and that proposed, while the saving in time would be found to be very great indeed.

Mr. CRAMPTON. I have a desire to see this method investigated because it seems to offer a very favorable modification, but I will mention a theoretical objection which has not been noticed, namely, that alkalis have some action upon glycerine. What would be the extent of this action, and what compounds would result therefrom, I do not know, but I think this matter should be investigated in connection with the proposed method.

Mr. PENNY. I can see no objection to the point just mentioned. The method might, of course, be tested by blank experiment. This method does away with the water bath and steam bath altogether, the digestion being done over a lamp, and not necessarily lasting more than three to five minutes. It is unnecessary to dissolve the fatty acids before distillation; they will dissolve themselves in the process of distillation.

The PRESIDENT. I am very favorably impressed with the modification at first sight.

Mr. Penny's motion was then put and carried.

Mr. JORDAN. I move that the method adopted for the determination of total nitrogenous matters in milk be reconsidered. Carried.

Mr. JORDAN. I desire to call attention to the fact that in the determination of total nitrogenous matters, in order to determine the amount of casein, albumen, etc., it is necessary to multiply by a certain factor. I have found, in conversation with certain gentlemen present, that all are not using the same factor. Some are using 6.33; others, 6.25. In order to secure uniformity, I move that 6.33 be adopted, as I think it nearer correct than 6.25.

Mr. WOODS. This matter is one of importance, but I do not know enough of the composition of the albuminoids to enable me to form an opinion as to whether a change would be desirable. I think it would be well to retain the old number, 6.25, unless some figures on the analysis of albuminoids or casein are presented for the guidance of the association.

Mr. BABCOCK. The number of the factor for albuminoids of milk, as given in *Blythe's Foods*, is 6.33, and this is the general factor used by European analysts.

Mr. JORDAN. I suggest as an amendment to the motion that 6.25 be retained, and a committee be appointed to take the matter into consideration and report at the next meeting. Carried.

Mr. BABCOCK. Has any of the members present had any experience in the analysis of cheese, especially in the determination of water in cheese? I have found the results, in a large number of determinations, very unsatisfactory. If the water is determined by heating at 100°, a constant weight can not be obtained, even if the heating be contin-

ued for a week or ten days; there is a constant loss, so that the percentage as determined at 100° C. would vary according to the length of time it is dried, and the results obtained by heating are from 2 to 7, 8, and even 10 per cent higher than those derived by drying to constant weight over sulphuric acid. I call attention to the fact that the association has prescribed no method for determining the percentage of water in cheese, and think this subject should be taken into consideration during the ensuing year by the reporter upon dairy products.

Mr. PENNY. I suggest that the association decide upon what should be considered a standard butter. I have found, in making analyses for creamery men, that they wanted results expressed in butter rather than in butter fat, and have been puzzled to know just what was a fair average of the composition of butter, whether 85 per cent of fat, or 80 per cent, or lower.

Mr. VAN SLYKE. My recollection is that the Society of Public Analysts, of England, has fixed the standard of butter at 85 per cent of butter fat.

Mr. COOKE. I think 82 per cent would come about as close as anything that could be obtained in the composition of the better grades of butter in the United States. In regard to the determination of moisture in cheese, the following method has been settled upon at the Vermont Station:

> Take a fairly good quality of linen and make a little bag about an inch wide and 2 inches long. Fill this with the portions of the cheese to be analyzed, say 3 to 4 grams. Put it in a Soxhlet extractor, and digest it for three or four hours; then dry for the determination of the moisture. The very thorough exhaustion of the fat by ether leaves the casein in a condition in which the water dries out fairly well.

The results by this method would certainly not be too high; if anything, they would be low. We use the same method also in determining the casein in cheese, taking the little sacks and putting them into a Kjeldahl digestion flask, for the nitrogen determination, digesting the sack and all.

The results on fat obtained by this method agree very closely with those obtained by drying out first and then extracting, and they also agree very well with those obtained by putting the cheese into a bottle and treating by the Beimling method.

Mr. BABCOCK. Mr. Short, chemist to the State Dairy Commission of Wisconsin, has been working some time upon this matter of determining the moisture in cheese, and got fairly uniform results by digesting the sample of cheese in absolute alcohol for some time, and then driving off the alcohol and drying out the fat product. I offer this merely as a suggestion. By the addition of small quantities of absolute alcohol the water can be driven off from the cheese at a comparatively low temperature without the cheese apparently undergoing any decomposition, but heating in a water bath at 100° would give too high results for the water, either through the dissociation of some of the products other

than fat in the cheese, or for some other unknown reason, and constant results can apparently not be obtained in that way.

The PRESIDENT. It is customary to refer such questions by special vote to the reporter.

Mr. VAN SLYKE. I move that the matter of the examination of butter and cheese, and also the matter mentioned by Mr. Penny, be referred to the reporter on dairy products for the ensuing year. Carried.

Mr. Woods presented the following report:

FEEDING STUFFS LOW IN CARBOHYDRATES.

By C. D. WOODS.

The reporter on feeding stuffs rich in carbohydrates decided to send out two samples for analysis, so that in order not to increase the work of the stations too much it was decided to send out only one sample poor in carbohydrates. With this understanding an invitation was sent to all of the experiment stations and the Department of Agriculture, to coöperate in this work. The Department of Agriculture and twenty-four of the stations accepted the invitation, but only fifteen reports have been received.

Of the materials which seem to be most deficient in carbohydrates, cotton seed was selected as being the more desirable. Quite a large quantity of a good grade cotton-seed meal was sifted, and all that would not pass through a round hole 1 mm. in diameter was rejected. The sample taken for analysis consisted of that part which passed through the sieve. Inasmuch as this analysis was not intended to find out the composition of cotton seed, but as a test of methods, the reporter saw no objection to this way of preparing the sample, especially as the percentage of fiber was thereby considerably reduced. The sifted material, about a half bushel, was placed upon a large sheet of paper and thoroughly sampled, and from this successive portions were taken until a subsample of about a quart was obtained. From this subsample two small portions were taken to form part of two samples for analysis by ourselves. The subsample was then again sampled, and two 4-ounce bottles filled by taking small successive portions of the material. The remainder of the sample was then returned to the large sample, which was again thoroughly sampled and another subsample selected, from which portions for analysis for ourselves were taken as before, and two more bottles filled. In this way by successive sampling enough bottles were filled for the use of those who had expressed their willingness to take part in the work. The bottles were carefully stoppered with sound, close-fitting corks, and then sealed with paraffin. It is believed that in this way the samples sent to the different analysts could not have differed materially the one from the other. The two samples obtained by taking small portions from each of the samples prepared for the analysts, were analyzed by Mr. Gibson, of the Storrs Station, with the following results:

Sample.	Water.	Ash.	Fat.	Fiber.	Nitrogen.	
					Total.	Alb.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
No. 1	7.20	7.62	9.70	4.42	7.18	6.93
No. 2	7.21	7.62	9.69	4.45	7.18	6.94

The close agreement of the analyses of duplicate samples would seem to justify the conclusion that the differences found by different analysts were not due to the variability of samples.

The results of the analyses calculated on the water basis found by each analyst are given in the following table. There are also given the minimum and maximum and average of each ingredient.

COTTON-SEED MEAL.

[Results of analyses.]

Number.	State.	Analyst.	Water.	Ash.	Fat.	Fiber.	Nitrogen.	
							Total.	Alb.
			Per ct.	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*
2	Connecticut, State	A. L. Winton, jr	8.23	7.62	9.50	4.38	7.12	6.99
6	Arkansas	G. L. Teller	7.98	7.61	9.72	5.18	7.12	6.69
8	Indiana	H. A. Huston	7.64	7.74	9.55	4.85	7.14	6.90
9	West Virginia	Rudolf de Roode	7.91	7.69	9.57	5.30	7.02	6.67
11	Alabama	N. T. Lupton	6.67	7.76	9.78	4.68	7.02	6.53
12	Mississippi	L. G. Patterson	7.59	7.58	9.55	5.15	7.14	6.94
15	New Jersey	L. A. Voorhees	6.72	7.62	9.46	4.56	7.19	6 94
16	Kansas	J. T. Willard	5.97	7.72	9.39	4.57	7.10	6.81
17	New York, State	R. B. Armstrong	7.61	7.88	9.50	*6 34	7.20	7.14
18	Wisconsin	F. W. Woll	7.43	7.65	9.42	5.25	7.20	7.03
20	Maryland	H. J. Patterson	5.70	7.52	10.25	4.77	7.16	6.90
22	New York, Cornell	Harry Snyder	7.66	7.46	9.40	4.72	7.16	6.71
23	Department of Agriculture	Aug. E. Knorr	7.46	7.86	9.44	4.61	7.17	7.03
24	Illinois	E. H. Farrington	7.70	7.55	10.25	5.02	6.90	6.55
	Connecticut, Storrs	H. B. Gibson	7.21	7.62	9.70	4.44	7.18	6.94
	Minimum		5.70	7.46	9.40	4.38	6.90	6.53
	Maximum		8.23	7.88	10.25	6.34	7.24	7.14
	Average		7.29	7.65	9.63	4.77	7.12	6.85

* Not included in average.

COTTON-SEED MEAL.

[Differences above and below average.]

Station.	Water (per cent).		Ash (per cent).		Fat (per cent).		Fiber (per cent).		Nitrogen.			
									Total (per cent).		Album. (per cent).	
	+	—	+	—	+	—	+	—	+	—	+	—
Connecticut, State	.94	.....	.....	.03	.....	.13	.....	.39	.00	.00	.14	.....
Arkansas	.69	.....	.....	.04	.09	.....	.41	.....	.00	.00	.....	.16
Indiana	.35	.....	.09	.....	.....	.08	.08	.....	.02	.....	.05	.....
West Virginia	.62	.....	.04	.....	.....	.06	(*)	(*)	.....	.10	.....	.18
Alabama	.....	.62	.11	.....	.15	.....	.....	.09	.....	.10	.....	.32
Mississippi	.30	.....	.....	.07	.....	.08	.38	.....	.02	.....	.09	.....
New Jersey	.....	.57	.....	.03	.....	.17	.....	.21	.07	.....	.09	.....
Kansas	.....	1.32	.07	.....	.....	.24	.....	.20	.....	.02	.....	.04
New York, State	.32	.....	.23	.....	.....	.13	(*)	(*)	.08	.....	.19	.....
Wisconsin	.14	.....	.00	.00	.....	.21	.48	.....	.08	.....	.18	.....
Maryland	.....	1.59	.....	.13	.62	.....	.....	.00	.04	.....	.05	.....
New York, Cornell	.37	.....	.....	.19	.....	.23	....	.05	.04	.....	.....	.14
Department of Agriculture	.17	.....	.21	.....	.....	.19	.....	.16	.05	.....	.18	.....
Illinois	.41	.....	.....	.10	.62	.....	.23	.....	.....	.22	.....	.30
Connecticut, Storrs	.....	.08	.....	.03	.07	.....	.....	.33	.06	.....	.09	.....

* Not included.

NOTES BY ANALYSTS.

WATER.

Maryland.—The moisture was determined in the ordinary steam bath without the use of hydrogen.

Kansas.—In the determination of water the pure dry hydrogen was passed through, not simply over, the substance. I am satisfied that four hours is not sufficient to effect the drying in all cases. After heating for the specified four hours and weighing, I heated the sample four hours longer, and the result was a total loss of 6.44 per cent, instead of 5.97 per cent, as given above for moisture.

Mississippi.—There was no loss of water (moisture) after heating for four hours.

Alabama.—For determination of water, four hours in a current of dry hydrogen was not long enough for complete drying.

FATS—ETHER EXTRACT.

New York State.—Undried substance extracted with anhydrous alcohol-free ether gave 9.46 per cent fat.

New York, Cornell.—German ether, labeled "distilled from sodium" was used. It was again treated with sodium, and distilled off only as wanted. While the extraction was going on, the mouth of the return flow condenser was closed by a chloride of calcium tube.

New Jersey.—Ether extract dried in air.

Maryland.—Ether extract purified by animal charcoal, 9.03 per cent.

Illinois.—Ether extract in air, dry sample, 10.31 per cent.

Kansas.—Ether extract obtained by use of absolute ether freshly redistilled from phosphorous anhydride. A glass siphoning apparatus was used.

FIBER.

Connecticut State Station.—No inverted condenser was used in fiber determinations as we boil, so that the loss of water is extremely small. The fiber was filtered and washed on paper, for it was absolutely impossible to filter it in a Gooch filter.

New York, Cornell.—Glasswool and asbestos filter was used.

West Virginia.—Considerable difficulty was experienced in the fiber determinations, and the results as returned were marked with an interrogation point.

Kansas.—Asbestos filter was used.

Arkansas.—A linen filter was used for the first filtration, and Gooch crucible for the second. The alkali solution was used first.

As perhaps was to have been expected, the greatest difference in results occurs in the determination of water, where there is a range of 2.5 per cent from the highest to the lowest. As will be noticed in the table of differences, the average is probably made at least one-half per cent too low; for if we should proceed to reject the highest and lowest, those that remain would raise the percentage by at least that much. The differences in percentages of ash are, with two or three exceptions, not great. The table of differences would seem to indicate that the average, as given in the composition table, is a little too high. With the exception of the analyses from the Maryland and Illinois stations, the percentages of fat are quite concordant. The table of differences would seem to indicate that the average as given is too high by two-tenths or three-tenths of a per cent. The percentage of fiber was also probably somewhat too high. That of the New York State Station was not taken into account in the average, because it was so much higher than that reported from any other station; and that of West Virginia was also excluded, as the analyst placed an interrogation point after his results, which seemed to imply lack of confidence in the determinations. With one exception, the differences in percentages of total nitrogen are not very great, and the differences in albuminoid nitrogen are somewhat greater. On the whole, the results seem to be much more satisfactory than any that have been presented in previous years, and it seems to the reporter as though this greater uniformity may be partly accounted for in a uniform method of estimation.

Mr. WOODS. I do not desire to make any recommendations of methods or changes until after the report on foods and feeding stuffs rich in carbohydrates has been presented, as all the changes which are to be suggested would be incorporated in that report.

The president presented the following papers:

DETERMINATION OF FATS IN FODDERS BY DIRECT WEIGHING AND BY LOSS OF WEIGHT OF THE SUBSTANCE.

By HARRY SNYDER.

The present official method for the determination of fats in fodders requires that the substance should be dried in a current of dry hydrogen, at the temperature of boiling water, for four hours, to remove all of the moisture; the dried substance is then extracted with ether, and the extract dried to constant weight under the same conditions.

In order to meet all of these requirements, Prof. Caldwell described in 1888 "A new apparatus for drying substances in hydrogen, and for the extraction of the fat." A reference to the cut and the description[1] will show that these tubes serve the double purpose of glass-stoppered weighing tubes, as well as drying tubes. This apparatus was made use of in the work described in this paper in the manner indicated in the bulletin, except that the current of hydrogen was conducted in the opposite direction. This change was found to be preferable to prevent mechanical losses by particles of the substance being carried along with the current, and also to prevent the loosening of the asbestos filter from the metallic disk supporting it.

In the same bulletin Prof. Caldwell says: "When the extraction is completed there seems to be no reason why the amount extracted can not be determined by weighing the dried contents of the tube as well as by weighing the substance extracted, and the drying in hydrogen is much more easily managed in the former case than in the latter, but I have not yet had time to test this variation of the method."

At the seventh annual convention of this association Mr. Anderson reported on two substances that the percentage of fat by loss of weight was a little larger than by weighing the extract. This he accounted for by saying that a portion of the fat was volatilized during the extraction.

In order to obtain some idea as to the favorable or unfavorable conditions for the loss of volatile fats during the extraction process, a short thermometer, 12½ cm. long, was embedded in the fodder tube when it was ready for extraction. The thermometer was of such a length that it would be entirely inclosed in the extraction apparatus. Another thermometer was inserted in the ether flask. A number of temperature determinations were made in this way. The temperature of the substance in the extraction tubes ranged from 33° to 35° C., the temperature of the contents of the ether flask registered 41° to 43° C.

The substance, before and after extraction, and the fat were all dried at the temperature of boiling water, and it would hardly seem possible that this lower and intermediate temperature of 43° would be the cause of a loss of a portion of the extract by volatilization or dissociation. During the extraction the fat is in solution, and the conditions as to mechanical state and temperature are less favorable for volatilization than in either the previous or subsequent drying at the temperature of boiling water.

A number of comparisons were made by the two methods. The tubes were dried first the official four hours to remove the moisture, but the data obtained in this way were confusing; with some substances the agreement was close, but with others not so, there being a general tendency for higher results by the loss of weight. The work was repeated on the same substances in the following manner: Instead of drying four hours, the drying was continued until the duplicate weighings of the tubes agreed within 1 milligram. This required a variable length of time which always exceeded

[1] Bulletin No. 12, Cornell Univ. Agr. Ex. Station.

four hours, and in the case of gluten meal reached six hours. When the fats were determined by loss after drying in this way, the agreement was much more satisfactory.

From the results thus obtained it appears that the first drying of four hours was not sufficient to remove all of the moisture, and the drying after the extraction removed the moisture left on the first drying, so that the loss of weight of the tubes also included a variable amount of moisture with the ether soluble matter. Hence the larger results by loss of weight.

In the following table the results on the same line are from the same weighed portion. The tubes were dried in hydrogen both before and after the extraction, except that tube c in samples No. 6, 7, and 8, and tube b of No. 11, were dried in air after the extraction.

Fats.

Sample.		By loss of weight of extraction tube.	Average.	By weighing of ether extract.	Average.
		Per cent.	*Per cent.*	*Per cent.*	*Per cent.*
1. Brewers' grains	*a*	7.34	7.35	7.26	7.36
	b	7.35		7.47	
2. Timothy hay	*a*	2.61	2.55	2.59	2.55
	b	2.50		2.52	
3. Red-clover hay	*a*	3.31	3.22	3.27	3.22
	b	3.15		3.18	
4. Gluten meal	*a*	(10.80)	11.16	11.24	11.18
	b	11.12		11.15	
	c	11.20		11.16	
5. Ensilage	*a*	2.22	2.25	2.19	2.25
	b	2.28		2.32	
6. Cotton seed meal*	*a*	9.35	9.39	9.33	9.40
	b	9.38		9.45	
	c	9.44		9.42	
7. Corn meal*	*a*	4.04	3.93	3.92	3.93
	b	3.92		4.04	
	c	3.85		3.85	
8. Ship stuff*	*a*	4.31	4.53	4.37	4.50
	b	4.72		4.67	
	c	4.56		4.46	
9. Ensilage	*a*	2.41	2 27	2.45	2.36
	b	2.12		2.28	
10. Ensilage	*a*	3.20	2.97	3.20	2.97
	b	2.73		2.73	
11. Ensilage	*a*	2.60	2.55	2.57	2.61
	b	2.50		2.66	
12. Ensilage	*a*	Lost.	2.43	2.41	2.42
	b	2,43		2.43	

* Committee sample.

The total average by weighing the fat was 0.006 per cent higher than by the loss of weight; a difference which is insignificant.

The tubes are more convenient for drying than flasks, and no additional or expensive drying apparatus is required, as for the flasks.

The drying of the tubes after the extraction can be done much more quickly and efficiently than drying the flasks, for the gas passes through the substance rather than over its surface, thereby requiring less hydrogen. More than this, the results show that the use of hydrogen in this second drying can be dispensed with.

The weighings of the flasks for the fats can be dispensed with.

On the whole, therefore, it is more economical as to time, apparatus, and hydrogen, to determine the fats by loss of weight of the substance, while there is no loss in accuracy.

AN ERROR IN THE PRESENT OFFICIAL METHOD FOR THE DETERMINATION OF ALBUMINOID NITROGEN; AND THE EFFECT OF THE PRESENCE OF METALS THAT ARE PRECIPITABLE BY POTASSIUM SULPHIDE IN THE DETERMINATION OF NITROGEN BY KJELDAHL'S METHOD.

By Harry Snyder.

In the determination of nitrogen by the Kjeldahl method directions are given for the addition of potassium sulphide to precipitate all the mercury from the solution, in order to prevent the formation of mercurammonium compounds which are not completely decomposed by soda solution.

The determination of albuminoid nitrogen according to the present official method requires the use of 0.7 to 0.8 gr. of copper hydroxide previous to the determination of the nitrogen by the Kjeldahl method.

The copper hydroxide is carried along in the determination, and is freely soluble in the sulphuric acid; no provision is made for its removal, and when the potassium sulphide is "added to precipitate all mercury in solution," copper sulphide is alike precipitated in the acid solution with the mercury. The copper present is more than enough to combine with all the potassium sulphide; the result is that neither metal is completely precipitated, and a large and variable portion of the mercury is left in the solution, and all the conditions are favorable for the formation of mercurammonium compounds. The work given to the potassium sulphide is doubled, and the object for which it is added is not attained.

In order to determine to what extent the mercury so left in the solution would affect the results, a number of determinations were started. To some the official "20 cc. of potassium sulphide" was added, and to others a quantity of the same solution sufficient to precipitate all the copper as well as all the mercury in solution. The large amount of precipitate formed in the latter case caused so much bumping that only one determination out of six could be completed.

In order to obviate this difficulty, the operation was carried on exactly as required by the official method, except that previous to the distillation the acid solution was made up to 200 cc. and divided into two 100 cc. portions. One portion was distilled with the relative official amount of potassium sulphide, and the second with enough of the same solution to complete the reaction. This gives a more satisfactory basis for the comparison of results, since all the conditions previous to the distillation are the same as to the degree of oxidation, and the amounts of mercury and copper in solution.

The following results were obtained as percentages of albuminoid nitrogen:

Ensilage.	50 cc. K_2S.	20 cc. K_2S.	Difference.
	Per cent.	*Per cent.*	*Per cent.*
No. 1	1.28	1.12	0.16
	1.25	1.14	0.11
No. 2	0.66	0.53	0.13
	0.66	0.60	0.06
No. 3	1.36	1.27	0.09
	1.20	1.15	0.05
No. 4	1.52	1.45	0.07
	1.54	1.45	0.09
No. 5	1.18	1.08	0.10
	1.23	1.12	0.11
Average			+0.096

Since all the conditions previous to distillation and addition of the potassium sulphide were the same in each portion as to the degree of oxidation and amounts of mercury and copper in solution, the constant larger proportion of nitrogen yielded with 50 cc. of potassium sulphide must be due to the incomplete precipitation of all the mercury in solution when only 20 cc. is added. The average difference in the ten portions amounted to nearly a tenth of 1 per cent. This is only a small amount, but when multiplied by the protein factor it amounts to over 0.60 per cent. Blank determinations with both 20 and 50 cc. portions of potassium sulphide showed no amount of nitrogen.

Some change must therefore be made in combining Stutzer's and Kjeldahl's methods. The following proportions of substance and solutions will give satisfactory results: Take 0.7 to 0.8 gr. of substance instead of 1 gr., use a quantity of the copper hydroxide and glycerine mixture, containing 0.5 to 0.6 gr. of the hydroxide, instead of 0.7 to 0.8 gr., and finally, 30 cc. of potassium sulphide instead of 20 cc.

Trials with these proportions on the committee samples of corn meal gave 1.56 and 1.55 per cent of albuminoid nitrogen; with the old proportions, and precipitating all the copper, 1.56 per cent.

Mr. WOODS. At the Missouri Station a study of the composition of the flesh of animals raised on the station farm has been undertaken, and I have a paper on this subject which has been sent by Dr. Schweitzer and Mr. Fox, which I ask to have incorporated in the proceedings under the head of materials not rich in carbohydrates.

The paper follows:

MEAT ANALYSIS—TESTING OF APPARATUS AND METHOD.

By P. SCHWEITZER and C. P. FOX.

(1) *Drying and water determination.*

The oven was a simple sheet-iron affair, 44 inches high and 6 inches square, furnished with ten removable wire-gauze trays; it was heated by a supply of hot air conducted through two copper tubes, one on each side, into a shallow chamber at the bottom, from which the heated air passed successively through the perforated trays into the chimney at the top, which could be lengthened to 2 feet or more to increase the draft. The apparatus worked very satisfactorily, the temperatures indicated below being respectively those of the top and of the bottom shelf.

The meat of a beefsteak was put several times through a sausage cutter, well mixed, and three portions weighed off—two on watch glasses, the third on the screen of the drying oven direct—and carefully dried at low temperatures, and weighed from time to time. The results were as follows:

[The loss by drying is indicated in grams and per cent.]

	Temperature.	1.	2.	3.
	Degrees C.			
Original weight of support and meat		129.8 gr	81.2 gr	111.4 gr.
Weight of support		29.8 watch glass.	31.2 watch glass.	61.4 screen.
Weight of meat		100.0 gr	50.0 gr	50.0 gr.
First hour—Top shelf	38	4.6 gr	2.6 gr	5.3 gr.
Bottom shelf	53	4.6 per cent.	5.2 per cent.	10.6 per cent.
Second hour—Top shelf	38	3.0 gr	1.8 gr	6.1 gr.
Bottom shelf	53	3.0 per cent.	3.6 per cent.	12.2 per cent.
		7.6 per cent.	8.8 per cent.	22.8 per cent.

	Temperature.	1.	2.	3.
	Degrees C.			
Third hour—Top shelf	38	2.4 gr	1.5 gr	3.8 gr.
Bottom shelf	50	2.4 per cent.	3.0 per cent	7.6 per cent.
		10.0 per cent.	11.8 per cent	30.4 per cent.
Fifteenth hour (over night)—Top shelf	39	15.5 gr	8.2 gr	14.4 gr.
Bottom shelf	52	15.5 per cent.	16 4 per cent.	28.8 per cent.
		25.5 per cent.	28.2 per cent	59.2 per cent.
Sixteenth hour—Top shelf	42	2.5 gr	1.3 gr	1.1 gr.
Bottom shelf	55	2.5 per cent.	2.6 per cent.	2.2 per cent.
		28.0 per cent.	30.8 per cent.	61.4 per cent.
Seventeenth hour—Top shelf	47	2.3 gr	1.5 gr	0.8 gr.
Bottom shelf	59	2.3 per cent	3.0 per cent	1.6 per cent.
		30.3 per cent	33.8 per cent.	63.0 per cent.
Eighteenth hour—Top shelf	42	1.9 gr	1.2 gr	0.8 gr.
Bottom shelf	51	1.9 per cent.	2.4 per cent.	1.6 per cent.
		32.2 per cent	36.2 per cent.	64.6 per cent.
Nineteenth hour—Top shelf	39	2.1 gr	1.1 gr	0.5 gr.
Bottom shelf	51	2.1 per cent.	2.2 per cent	1.0 per cent.
		34.3 per cent.	38.4 per cent	65.6 per cent.
Twentieth hour—Top shelf	42	1.8 gr	0.9 gr	0.3 gr.
Bottom shelf	53	1.8 per cent.	1.8 per cent	0.6 per cent.
		36.1 per cent.	40.2 per cent.	66.2 per cent.
Thirty-second hour (over night)—Top shelf	35	15.2 gr	7.0 gr	1.3 gr.
Bottom shelf	51	15.2 per cent	14.0 per cent	2.6 per cent.
		51.3 per cent.	54.2 per cent.	68.8 per cent.
Thirty-third hour—Top shelf	44	2.9 gr	1.5 gr	0.3 gr.
Bottom shelf	64	2.9 per cent.	3.0 per cent.	0.6 per cent.
		54.2 per cent.	57.2 per cent.	69.4 per cent.
Thirty-fourth hour—Top shelf	43	1.8 gr	0.6 gr	
Bottom shelf	63	1.8 per cent.	1.2 per cent.	
		56.0 per cent.	58.4 per cent.	69.4 per cent.
Thirty-fifth hour—Top shelf	45	2.0 gr	0.9 gr	0.1 gr.
Bottom shelf	64	2.0 per cent.	1.8 per cent	0.2 per cent.
		58.0 per cent.	60.2 per cent.	69.6 per cent.
Thirty-sixth hour—Top shelf	41	1.4 gr	0.5 gr	0.1 gr.
Bottom shelf	57	1.4 per cent.	1.0 per cent.	0.2 per cent.
		59.4 per cent.	61.2 per cent.	69.8 per cent.
Thirty-seventh hour—Top shelf	43	0.8 gr	0.4 gr	0.1 gr.
Bottom shelf	63	0.8 per cent.	0.8 per cent.	0.2 per cent.
		60.2 per cent.	62.0 per cent.	70.0 per cent.
Thirty-eighth hour—Top shelf	47	1.0 gr	0.3 gr	
Bottom shelf	52	1.0 per cent	0.6 per cent.	
		61.2 per cent	62.6 per cent.	70.0 per cent.
Forty-sixth hour (over part of night)—Top shelf	39	6.0 gr	1.7 gr	0.2 gr.
Bottom shelf	60	6.0 per cent.	3.4 per cent.	0.4 per cent.
Total loss in water		67.2 per cent.	66.0 per cent.	70.4 per cent.
Weight of dried meat (*a*)		32.8 gr	17.0 gr	14.8 gr.

The substance was now carefully powdered and put into a bottle, and weighed with the following results:

	1.	2.	3.
Dried meat in bottle (*b*)	31.5 gr.	16.2 gr	14.6 gr.
Loss by powdering, etc	1.3 gr.	0.8 gr.	0.2 gr.
	1.3 per cent.	1.6 per cent.	0.4 per cent.

Two gr. of the powdered meat dried at 110° C. lost as follows:

	1.	2.	3.
	0.2001 gr.	0.1782 gr.	0.0410 gr.
	10.00 per cent	8.91 per cent.	2.05 per cent.

On calculating this loss for the whole of the dried meat left and powdered and bottled, the results were as follows (*a*):

	1.	2.	3.
	2:32.8=0.2001:x; x=3.28 gr.		
	2:17.0=0.1782:x; x=1.50 gr.		
	2:14.8=0.0410:x; x=0.30 gr.		
Total water in grams	70.48	34.50	35.50
Total water in per cent	70.48	69.00	71.00

Or, using the weight of meat actually bottled (*b*):

	1.	2.	3.
	2:31.5=0.2001:x; x=3.15 gr.		
	2:16.2=0.1782:x; x=1.44 gr.		
	2:14.6=0.0410:x; x=0.30 gr.		
Total water in grams	70.35	34.44	35.50
Total water in per cent	70.35	68.88	71.00

(*b*) *Determination of fat.*

This was determined in 2 gr. of the dried meat, evaporating the ether extract, drying and weighing the fat directly, as follows:

	1.	2.	3.
	0.4022 gr.	0.4166 gr.	0.4182 gr.

Calculating this weight for the whole of the dried meat (*a*):

	1.	2.	3.
Total fat in grams	6.5960	3.5411	3.0946
Total fat in per cent	6.5960	7.0822	6.1892

Or, for the meat bottled (*b*):

	1.	2.	3.
Total fat in grams	6.3346	3.3744	3.0528
Total fat in per cent	6.3346	6.7488	6.1056

The indirect determination of fat was likewise made by carefully drying and weighing the extracted material, giving the following losses:

	1.	2.	3.
	0.4116 gr.	0.4174 gr.	0.4264 gr.

Calculating these weights for the whole of the dried meats (*a*):

	1.	2.	3.
Total fat in grams	6.7502	3.0479	3.1554
Total fat in per cent	6.7502	7.0958	6.3108

Or, for the meat bottled (*b*):

	1.	2.	3.
Total fat in grams	6.4827	3.3809	3.1127
Total fat in per cent	6.4827	6.7618	6.2254

(*c*) *Determination of nitrogen* (*Kjeldahl*).

In 2 gr. of dried meat:

	1.	2.	3.
	0.2045 gr.	0.2087 gr.	0.2291 gr.

For the whole of the dried meat (*a*):

	1.	2.	3.
Total nitrogen in grams	3.3538	1.7739	1.6953
Total nitrogen in per cent	3.3538	3.5478	3.3906

For the meat bottled (*b*):

	1.	2.	3.
Total nitrogen in grams	3.2209	1.6905	1.6724
Total nitrogen in per cent	3.2209	3.3810	3.3448

Considering the nitrogen to furnish the albuminoids present, by multiplying with 6.25 the result would be as follows:

	1.	2.	3.
	Per cent.	*Per cent.*	*Per cent.*
Albuminoids (*a*)	20.9612	22.1737	21.1912
Albuminoids (*b*)	20.1306	21.1312	20.9050

(*d*) *Recapitulation and composition of meat.*

	1.	2.	3.
	Per cent.	*Per cent.*	*Per cent.*
Water (*a*)	70.480	69.000	71.000
Fat	6.596	7.082	6.189
Albuminoids	20.961	22.174	21.191
Ash, etc	1.963	1.744	1.620
	100.000	100.000	100.000

Or, as in previous calculations:

	1.	2.	3.
	Per cent.	*Per cent.*	*Per cent.*
Water (*b*)	70.350	68.880	71.000
Fat	6.335	6.749	6.106
Albuminoids	20.131	21.131	20.905
Ash, etc	1.884	1.640	1.589
Loss	1.300	1.600	0.400
	100.000	100.000	100.000

(*e*) *Water in lean-meat fiber.*

Supposing the fat not to contain any water, the whole of the latter must then belong to the lean part of the meat, amounting, exclusive of fat, ash, etc., to the following percentages:

	1.	2.	3.
	Per cent.	*Per cent.*	*Per cent.*
(*a*)	77.07	75.68	77.01
(*b*)	77.75	76.52	77.25

(*f*) CONCLUSION.

The results under (*a*), by distributing the loss, are considered the more trustworthy and reasonable, and will be given in the future investigation of "experimental cattle." The deviations under No. 2 are explainable. Intending to use three times 100 gr. of meat, the supply, after cutting, was found to be insufficient; 50 gr. each was then taken for No. 2 and 3, but even this amount fell short in No. 2, and the portions remaining in the sausage cutter and containing some lumpy and cartilaginous matter, had to be used to make up the 50 gr.

Mr. Knorr presented the following report on feeding stuffs rich in carbohydrates:

REPORT ON CATTLE FOODS RICH IN CARBOHYDRATES.

By Aug. E. Knorr.

About the middle of last November a circular letter was issued soliciting coöperation on the part of the various State Experiment Stations in the comparative analytical work on highly carbohydrated cattle foods for the season 1890–'91.

This invitation was accepted by twenty-nine different chemists, and in the course of the year reports from fifteen of these were received.[1]

On December 1 two samples, one of ship stuff and one of yellow corn meal, were distributed. Accompanying these samples were detailed working instructions according to the methods adopted at the last meeting of the association, and also another circular letter stating the purpose of the reporter to perform a control analysis on each sample sent out. Following is the text of this letter:

U. S. Department of Agriculture,
Division of Chemistry,
Washington, D. C., December 1, 1890.

Dear Sir: I send you to-day two samples of cattle foods, as per circular letter of November 15.

One of the samples is a heavy ship stuff, and the other a yellow corn meal, and they are labeled Nos. 1 and 2, respectively. A portion of each specimen sent out has been retained in this laboratory, duly marked with the name of station where the remainder was sent, and it is my intention to perform a control analysis of the same on the exact date that the work is done at the corresponding station.

By careful comparison of the various data collected, I hope that we may in this manner be enabled to determine the cause of any considerable discrepancies in the figures obtained, should any such be reported. I think that this object can be attained if the cause of the variation of analyses in former years is to be sought, either in the difficulty or impossibility of uniform sampling, in the difference in time of performing analysis, or if it is owing to the personal error. On the other hand, if our methods are at fault, we may also obtain useful hints to guide us in future experiments.

The analyses may be made entirely at the leisure of each analyst; and the reporter only begs that the results be sent a reasonable length of time before the next convention, which it is proposed to hold early in September, so that he may not be too much pushed at the last moment in collecting the various data.

You will see the importance of notifying your reporter of the exact date on which you wish to do the work; hence, I beg you would advise me about two weeks before the time, so that I may make ample provisions with my own work here.

The samples have all been carefully sealed at the same time, and it is essential that when once opened the analysis should at once be made and proceeded with without interruption until all the official determinations are complete. Then, should the individual analysts desire to make further experiments on new and original lines, the reporter would be very glad indeed to include their work in his report at the next meeting; and he will hold himself ready to furnish an additional portion of the corresponding sample sent to each station, and this has been placed aside for this parparticular purpose.

The reporter, at the last meeting, especally recommended that Patterson's method of purification of ether extract with animal charcoal be tested, and that for comparison also ether extracts of the original samples be made without previous drying in hydrogen.

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[1] Since the reading of this report two additional reports have been received and embodied in the general tabular statements of analyses.

I enclose report blanks and circular containing full description of methods as adopted at the recent convention. I beg that the directions therein contained be followed to the letter, and that where variations and alternate methods are adopted, the reporter be furnished with a detailed statement of the *modus operandi*. I would finally suggest that before weighing out samples, the bottles containing the specimens be carefully rolled for a few minutes in order thoroughly to shake up any heavier particles that might have settled to the bottom owing to the continual shaking in one position that probably took place while on the road.

Respectfully,

AUG. E. KNORR,
Assistant Chemist.

In accordance with this plan, duplicate check samples were retained in the laboratory of the U. S. Department of Agriculture, the control analysis being performed at the proper time in each case, except that owing to pressure of other business, nitrogen determinations were omitted in the last two samples. I desire in this place to acknowledge the valuable aid received from Mr. T. C. Trescot, who performed all of the nitrogen determinations.

It may be remembered that at the last meeting of the association a division of the cattle foods was made into two groups, those rich in fat and nitrogen on the one hand, and those rich in carbohydrates on the other, individual reporters being appointed for each division. Bearing on this subject your reporter is in receipt of a letter dated February 4, from Dr. G. C. Caldwell, of Cornell University, who reported on cattle foods at the last meeting. I quote the following passage from this letter: "One matter is not clear to me. The special reason why the appointment of an additional reporter was recommended and ordered by the association was, if I am not mistaken, that the same method of analysis would not work for feed stuffs rich in carbohydrates, and for those rich in protein and fat, and I understood that the method heretofore followed must be modified for starchy feed stuffs like milling products. Notwithstanding this I have the impression that you intend to have the old method, that of last year, followed substantially. Wherein am I wrong in my understanding of the matter?"

Commenting on the same subject, Dr. Woods, of Middletown, Conn., the reporter on the nitrogenous group of cattle foods for this meeting, wrote me in a letter of October 2: "But I still fail to take in the proposed division of work between the two reporters. By your having starchy fodders is it intended to study in them starch, sugar, etc., as such only, or only to test the methods of fodder analysis on that class of fodders?"

In preparing my work I was at once confronted with the fact, that though the division of the cattle foods into two distinct classes had been effected, no provisions whatever had been made concerning the methods to be employed on the group of starchy fodders. As set forth on page 211, Chemical Bulletin No. 28, the official methods adopted for 1890–'91, apply to nonstarchy foods only, no official instructions or methods of any kind being laid down for the analysis of highly carbohydrated cattle foods.

What was there to be done under the circumstances? Your reporter could hardly take upon himself the preparation of a new working scheme applicable to the class assigned to him, and devoid of official basis or sanction. It was concluded that a conservative course would be the safest, and the work was consequently pursued according to the methods hitherto in use with the particular intention of subjecting them to a thorough test with reference to starchy foods, in the hope that this season's work might bring out the weak points and throw new light on the subject, on the strength of which the present methods might be modified or replaced by new ones.

The work was pursued on these lines, and no radical changes or innovations were attempted in the determinations of any of the constituents.

The two samples furnishing the subject for this year's investigation were obtained at one of our local feed stores, and the greatest possible pains were taken in their preparation.

About 3 kilos of the rough sample, as received from the dealer, were reduced in a small drug mill until every particle had passed through a sieve with meshes 1 mm. in diameter, as required by the official methods, and the iron from the mill was removed by means of a magnet. The sample, contained in a square box, and only about half filling the same, was then attached to the main belt shaft of the engine room and rolled for two days. This plan was suggested by Dr. Caldwell last year, and it is believed that in this manner mixing was performed most thoroughly. If it is possible to obtain an average sample of a substance of this nature, the samples distributed certainly represented such an average.

In undertaking to perform a control analysis on the various check samples retained in this laboratory, the reporter was far from assuming that his analyses would form an absolute standard of comparison, being fully aware of the fact that he was equally as liable to personal error as any of the individual coöperators. But, as in all likelihood the probable error of one person making a large number of analyses would vary within narrower limits than that of a number of analysts making the same number of analyses collectively, and, as it would moreover most probably be in the same direction from the true result, that is, either positive or negative, the standard of comparison may be considered a fair one for the purpose for which it was established.

In making up the general averages, only the averages of all the results obtained by the reporter were made use of. This was done in order to avoid giving his own results undue prominence over the figures obtained by the other analysts.

The official methods appear to have been faithfully carried out in all instances, noting a few unimportant variations, as given in the comments and statements of the several analysts below.

The following tables contain a statement of the analytical data reported, and as customary, the results are calculated to dry substance:

TABLE NO. 1.—*Ship stuff.*

State.	Analyst.	Date.	Moisture.	Ash.	Ether extract.	Crude fiber.	N. free extract.	Crude protein.	Nitrogen.	
									Total.	Alb.
			P. ct.	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*
Wisconsin	F. W. Woll	Jan. 26	11.26	5.67	5.31	10.23	58.67	20.12	3.22	2.98
New Jersey	L. A. Vorhees	Feb. 3	10.65	5.78	5.50	8.89	59.44	20.39	3.26	2.64
Massachusetts	W. H. Beal	Feb. 16	11.38	5.74	*6.15	8.94	58.86	20.31	3.25	2.97
Connecticut	H. B. Gibson (Storr's school).	Mar. 2	10.76	5.84	5.64	9.35	58.85	20.32	3.25	2.85
Illinois	E. H. Farrington	Mar. 9	11.87	*5.03	5.31	9.71	60.66	19.29	3.09	2.96
Connecticut	A. L. Winton (New Haven).	Mar. 16	*12.01	5.80	5.18	9.10	59.49	20.43	3.26	3.11
Michigan	R. C. Kedzie	do	11.65	6.11	5.63	9.61	59.09	19.56	3.13	2.50
New Hampshire	Chas. L. Parsons	Apr. 1	11.54	6.10	5.47	9.34	59.36	19.73	3.15	2.83
Alabama	J. T. Anderson	May 18	10.99	6.12	5.68	9.31	60.62	*18.27	2.92	2.41
South Dakota	Jas. H. Shepard	May 25	11.11	5.43	5.67	9.93	58.79	20.18	3.23	2.95
North Dakota	E. F. Ladd	June 1	[1]	5.55	*6.33	9.61	59.20	19.31	3.09	2.49
Kansas	J. T. Willard	June 15	*10.10	5.92	4.86	9.01	60.51	19.70	3.15	2.67
Maryland	H. J. Patterson	do	*10.10	5.49	*6.15	9.38	59.77	19.29	3.09	2.74
New York	H. Snyder	do	11.84	5.89	5.12	9.36	59.51	20.12	3.22	2.37
Do	R. B. Armstrong	June 20	11.48	5.98	5.51	*11.04	57.56	19.91	3.19	2.98
Indiana	H. A. Huston	July 1	11.55	5.98	5.10	9.72	59.26	19.94	3.19	2.94
Iowa	G. E. Patrick	do	11.21	[2]5.79	5.33	10.02	58.85	20.00	3.20	2.58
U. S. Department of Agriculture.	Aug. E. Knorr	Average of analyses.	11.55	5.99	5.33	9.51	59.08	20.09	3.22	2.96
Average			11.24	5.79	5.52	9.56	59.31	19.83	3.17	2.77
Difference between highest and lowest			1.91	1.09	1.47	2.15	3.10	2.16	.....	.....
*Eliminating widest results:										
Average			11.35	5.83	5.38	9.47	.....	19.92	.....	.....
Difference between highest and lowest			1.22	.69	.58	1.34	.....	1.14	.....	.....
This difference expressed in per cent of average.			10.75	11.84	10.78	14.15	.....	5.72	.....	.....
Widest result from average			.71	.43	.30	.75	.....	.67	.....	.....
This difference expressed in per cent of average			6.26	7.38	5.58	7.92	.....	3.36	.....	.....

[1] Results reported as calculated to dry substance without giving moisture.

[2] No ash determination having been reported, the average of the other analyses was taken.

TABLE NO. 2.—*Corn meal.*

State.	Analyst.	Date.	Moisture.	Ash.	Ether extract.	Crude fiber.	N. free extract.	Crude protein.	Nitrogen.	
									Total.	Alb.
			P. ct.	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*
Wisconsin	F. W. Woll	Jan. 26	11.65	1.52	*4.19	3.42	80.48	10.39	1.66	1.61
New Jersey	L. A. Vorhees	Feb. 3	10.89	1.52	4.60	2.79	80.11	10.98	1.76	1.67
Massachusetts	W. H. Beal	Feb. 16	11.83	1.54	*5.21	2.99	79.65	11.21	1.79	1.74
Connecticut	H. B. Gibson, (Storr's school).	Mar. 2	11.54	1.54	4.69	2.83	80.06	10.88	1.74	1.59
Illinois	E. H. Farrington	Mar. 9	11.88	1.52	4.59	2.89	80.27	10.72	1.71	1.66
Connecticut	A. L. Winton, (New Haven).	Mar. 16	12.32	1.56	4.65	*2.52	80.79	10.48	1.68	1.68
Michigan	R. C. Kedzie	...do	12.48	1.70	4.98	3.04	79.66	10.62	1.70	1.63
New Hampshire	Chas. L. Parsons	Apr. 1	11.88	1.48	4.69	2.98	80.47	10.38	1.66	1.65
Alabama	J. T. Anderson	May 18	*10.11	1.55	4.67	3.37	81.47	*8.94	1.43	1.19
South Dakota	Jas. H. Shepard	May 25	11.75	1.62	4.87	3.32	79.60	10.59	1.69	1.54
North Dakota	E. F. Ladd	June 1	[1]	1.45	4.99	2.97	81.84	*8.75	1.40	.....
Kansas	J. T. Willard	June 15	*10.16	1.59	4.40	2.83	80.89	10.29	1.65	1.58
Maryland	H. J. Patterson	... do	*10.00	1.32	*5.09	3.01	80.33	10.25	1.64	1.63
New York	H. Snyder	...do	12.07	1.22	4.53	2.91	80.26	11.08	1.77	1.77
Do	R. B. Armstrong	June 20	11.89	1.67	4.79	*4.38	77.03	*12.13	1.94	1.88
Indiana	H. A. Huston	July 1	12.09	1.55	4.57	3.17	80.07	10.67	1.71	1.60
Iowa	G. E. Patrick	...do	11.62	[2]1.52	4.34	3.13	80.47	10.54	1.68	1.58
U. S. Department of Agriculture.	Aug. E. Knorr	Average of analysis.	12.05	1.65	4.58	3.05	79.79	10.93	1.75	1.68
Average			11.54	1.53	4.70	3.09	80.70	10.54	1.67	1.62
Difference between highest and lowest			2.48	.48	.90	1.86	4.44	3.38	.....	.....
*Eliminating widest results:										
Average			11.85	1.53	4.68	3.04	.....	10.66	.....	.....
Difference between highest and lowest			1.59	.48	.58	.63	.....	.96	.....	.....
This difference expressed in per cent of average.			13.43	31.37	12.40	20.72	.....	9.00	.....	....
Widest difference from average			.98	.31	.38	.38	.....	.54	.....	.....
This difference expressed in per cent of average.			8.27	20.26	8.15	12.50	.....	5.01	.....	.....

An examination of these tables reveals at once that the analytical results obtained by the various chemists still lack uniformity, as in case of most of the constituents there is a difference of over 1 per cent, and in some instances 2 or 3 per cent, between the highest and lowest figures reported. A few of the data differing most widely from the general average have been marked with an asterisk, and, eliminating these figures from the general average, a new set of averages was made, showing a very marked increase of uniformity.

The figures thus discarded are twenty in number, making 11 per cent of the total number of 180 determinations reported. The widest range from the average is thus reduced to 0.98 per cent in case of the moisture in corn meal.

Although this still leaves considerable to be desired in the way of concordance in the analytical work, yet the improvement over the results of previous years is very marked, as may be gathered from a table comparing the work reported by Dr. Caldwell last year with this year's work.

[1] Results reported as calculated to dry substance without stating moisture.

[2] No ash determination having been reported, the average of the other analyses was taken.

TABLE No. 3.

	Moisture.		Ash.		Ether extract.		Crude fiber.		Nitrogen free extract.		Crude protein.	
	Extreme variation.	Expressed in per cent of average.	Extreme variation.	Expressed in per cent of average.	Extreme variation.	Expressed in per cent of average.	Extreme variation.	Expressed in per cent of average.	Extreme variation.	Expressed in per cent of average.	Extreme variation.	Expressed in per cent of average.
1889–'90.	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*
Hay	3.98	42.43	1.75	29.5	2.20	80.6	6.41	27.2	6.93	11.80	1.78	20.7
Wheat middlings	6.48	57.25	.42	42.4	1.19	58.9	.74	104.2	2 58	3.08	1.91	14.9
Linseed meal	5.94	64.22	1.26	21.4	1.12	11.7	2.41	32.5	8.92	24.20	5.50	13.5
1890–'91.												
Ship stuff	1.22	10.74	.69	13.12	.58	10.79	1.34	14.14	3.10	5.22	1.14	5.72
Corn meal	1.59	13.40	.48	31.37	.58	12.40	.63	20.73	4.44	5.50	.96	9.00

In Table No. 3 the first column in each case contains the difference between the highest and lowest figures reported, while the second column states this difference in per cent of average.

A tabular statement comparing the various data reported, with the corresponding control analyses performed in the Department laboratory, has been prepared in Tables No. 4 and 5.

TABLE No. 4.—*Ship stuff.*

	Moisture.		Ash.		Ether extract.		Crude fiber.		Crude protein.		Albuminoid nitrogen.	
	P. ct.	*C. P. ct.*	*P. ct.*	*C. P. ct.*	*P. ct.*	*C. P. ct.*	*P. ct.*	*C. P. ct.*	*P. ct.*	*C. P. ct.*	*P. ct.*	*C. P. ct.*
F. W. Woll	11.26	11.63	5.04	5.38	4.71	4.82	9.08	*8.92	17.85	*18.20	2.64	2.69
L. A. Vorhees	10.65	11.65	5.16	5.56	4.97	4.64	7.94	8.19	18.21	*18.38	2.36	2.66
W. H. Beal	11.38	11.66	5.09	5.24	*5.45	4.58	7.92	8.52	18.00	18.03	2.63	2.66
H. B. Gibson	10.76	11.35	5.21	5.11	5.03	4.61	8.34	8.18	18.13	17.68	2.54	2.63
E. H. Farrington	11.87	11.82	*4.44	*5.82	4.68	4.73	8.55	*8.85	17.00	17.68	2.61	2.63
A. L. Winton	*12.01	11.69	5.10	5.49	4.56	4.64	8.01	*8.00	17.97	18.03	2.74	2.59
R. C. Kedzie	11.65	11.48	5.40	5.48	4.97	4.71	8.49	8.47	17.28	17.85	2.21	2.56
Charles L. Parsons	11.54	11.78	5.40	5.14	4.84	4.67	8.26	8.52	17.45	17.85	2.50	2.63
J. T. Anderson	10.99	11.41	5.45	5.19	5.05	4.72	8.29	8.48	*16.26	17.50	2.17	2.58
James H. Shepard	11.11	11.31	4.83	5.19	5.04	4.74	8.83	8.44	17.94	17.68	2.62	2.57
E. F. Ladd[1]	.....	11.45	.....	5.06	.....	4.65	.....	8.56	.....	.....	.....	.....
J. T. Willard	*10.10	11.42	5.32	5.20	4.37	4.83	8.10	8.51	17.71	17.85	2.40	2.63
H. J. Patterson	*10.10	11.51	4.86	5.40	*5.53	4.76	8.43	*8.83	17.35	17.50	2.45	2.60
H. Snyder	11.84	11.47	5.20	5.23	4.51	4.78	8.25	8.55	17.74	17.85	2.09	2.60
R. B. Armstrong	11.48	11.59	5.29	5.29	4.88	4.82	*9.77	8.16	17.63	.....	2.64	.....
H. A. Huston	11.55	11.55	5.28	5.33	4.51	4.67	8.60	8.47	17.64	.....	2.60	.....
G. E. Patrick	11.21	11.36	5.12	5.38	4.73	4.82	8.90	8.44	17.76	.....	2.29	.....
Average	11.22	11.53	5.12	5.32	4.86	4.72	8.49	8.47	17.62	17.85	2.47	2.62
Extreme difference	1.91	.51	1.01	.76	1.16	.25	1.85	.92	1.95	.88	.65	.13
*Eliminating wide results	11.36	11.53	5.19	5.29	4.76	4.72	8.39	8.41	17.77	17.77	2.47	2.62
Extreme difference	1.11	.51	.62	.50	.67	.25	1.16	.39	1.21	.53	.65	.13

[1] Analyses were reported as calculated to dry substance, not stating actual results obtained.

TABLE NO. 5.—*Corn meal.*

	Moisture.		Ash.		Ether extract.		Fiber.		Crude protein.		Albuminoid nitrogen.	
	P. ct.	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*
F. W. Woll	11.65	12.20	1.34	1.46	*3.70	4.15	3.02	2.59	9.18	*10.15	1.42	1.49
L. A. Vorhees	10.90	12.08	1.36	1.45	4.10	4.09	2.49	2.90	9.78	9.94	1.49	1.52
W. H. Beal	11.83	11.78	1.36	1.41	*4.59	3.90	2.64	2.64	9.88	9.80	1.53	1.43
H. B. Gibson	11.54	12.04	1.36	1.35	4.15	3.96	2.50	2.60	9.63	9.63	1.41	1.47
E. H. Farrington	11.88	12.34	1.34	1.58	4.05	4.07	2.55	2.58	9.45	9.63	1.46	1.52
A. L. Winton	12.32	12.12	1.37	1.50	4.08	4.01	*2.21	2.57	9.19	9.80	1.47	1.47
R. C. Kedzie	12.48	11.87	1.49	1.61	4.36	4.13	2.66	2.67	9.29	9.45	1.44	1.48
Chas. L. Parsons	11.88	12.19	1.30	1.44	4.13	3.92	2.63	2.61	9.15	9.45	1.45	1.48
J. T. Anderson	10.11	11.81	1.39	1.38	4.20	4.02	3.03	2.63	*8.04	9.45	1.07	1.48
Jas. H. Shepard	11.75	12.12	1.43	1.35	4.29	4.04	2.93	2.83	9.35	9.45	1.39	1.47
E. F. Ladd[1]	.....	11.92	.....	1.37	.....	3.93	.....	2.90	.....	.....	.....	.....
J. T. Willard	10.16	11.88	1.43	1.51	3.95	4.03	2.54	2.84	9.25	*9.10	1.42	.....
H. J. Patterson	10.10	12.03	1.19	1.41	*4.58	3.99	2.76	2.65	9.22	9.45	1.47	1.46
H. Snyder	12.07	12.17	1.07	1.45	3.98	4.12	2.56	2.62	9.75	*9.10	1.56	1.43
R. B. Armstrong	11.89	11.98	1.47	1.53	4.22	4.01	*3.86	2.74	*10.69	.....	1.66	.....
H. A. Hustan	12.09	12.07	1.36	1.33	4.02	4.02	2.79	2.70	9.38	.....	1.41	.....
G. E. Patrick	11.62	11.60	1.35	1.53	3.84	4.06	2.77	2.62	9.33	.....	1.40	.....
Average	11.53	12.01	1.35	1.45	4.14	4.03	2.74	2.69	9.40	9.57	1.44	1.48
Extreme difference	1.37	.70	.42	.28	.89	.25	1.65	.33	2.65	.105	.59	.09
*Eliminating wide results	11.53	12.01	1.35	1.45	4.11	4.03	2.70	2.69	9.40	9.61	.....	.....
Extreme difference	1.37	.70	.42	.28	.52	.25	1.54	.33	.73	.49	.....	.....

The figures in this table are entered in chronological order, and a critical examination will show that none of the variations therein exhibited could by any stretch of the imagination be interpreted as being due to an influence of the time at which the analyses were made. In fact, this result is not unexpected, and it would have been surprising had this been different, for the samples had all been most carefully sealed at the same time, and were not opened and exposed to the air until the analyses were made. Neither can any of the differences be attributed to a difference in the various samples, for had such been the case, a wide result reported by any analyst would be followed by a similar wide result in the corresponding control analysis. This is by no means the case; on the very contrary, a high result being in some instances followed by a low result in the set of check determinations, and vice versa.

The only conclusion that can be drawn from this table, consequently, is that the variations reported are due to errors in manipulation, and to personal errors. That the methods themselves are not greatly at fault is shown by the marked improvement over the work of former years, encouraging the hope that another year may give us still greater accuracy.

As had been expected, this table also shows that the personal error of one individual making a large number of determinations is smaller than the error of a number of analysts making the same number of analyses collectively; and altogether it may be said that, aside from a few figures reported, the variations from the average have been reduced to within fairly reasonable limits by this year's work.

In addition to the analyses requested by the reporter, a number of coöperators furnished additional data and comments, and following are the statements made by them:

MOISTURE.

Alabama.—Four hours in a current of dry hydrogen, as directed, does not seem to produce complete drying, according to our opinion.

[1] Analyses were reported as calculated to dry substance, not stating actual results obtained.

(This experience of the Alabama Experiment Station is apparently borne out by the data reported, which fall considerably below the average.)

South Dakota.—Mr. Shepard gives a detailed description of the manipulations, and sends a sketch of the apparatus used by him. The hydrogen emerges from the apparatus through a tube dipping below sulphuric acid, which it was noticed soon became black, showing that something besides moisture is driven off, and this must introduce an error in both moisture and nitrogen free extract. This point will be recurred to below.

Kansas.—Mr. Willard does not believe that four hours' heating is sufficient. He is accustomed to heat one day (6 or 7 hours.)

Maryland.—Moistures were determined in simple steam oven without the use of hydrogen. (It will be seen by reference to the tables that these moisture determinations were more than 1 per cent below the average.)

New York (Cornell University).—Moisture determinations were made drying to constant weight, requiring five hours for the corn meal, and five and one half hours for the ship stuff.

TABLE NO. 6.—*Moisture determinations.*

[H. Snyder.]

	Corn meal.	Ship stuff.
	Per cent.	*Per cent.*
By drying to constant weight	12.45	12.03
By drying for four hours	12.07	11.86
Do	12.06	11.81

U. S. Department of Agriculture.—In the opinion of your reporter it is not a matter of indifference whether the hydrogen passes merely over the substance, or whether it has to pass through the same. In the arrangement employed at the Department laboratory and illustrated in Bulletin No. 28, page 100, the same current of hydrogen passes *through* each individual determination, and attention is directed to the very close agreement between the results obtained here and at the Indiana Experiment Station where a very similar apparatus was employed. The same blackening of the sulphuric acid was observed as had been noticed at the South Dakota Station, a strong odor of sulphurous acid being detected at the same time. This will be more fully discussed under ether extract.

TABLE No. 7.

[A. E. Knorr.]

Moisture.	Time of extraction.	Ether extract.			
		By weight.		By difference.	
Per cent.	*Hours.*	*Per cent.*	*Av'ge.*	*Per cent.*	*Av'ge.*
11.61	4	4.43	4.45	4.34	4.41
11.73		4.46		4.47	
11.73	8	4.45	4.49	4.45	4.44
11.70		4.53		4.43	
11.77	12	4.53	4.57	4.50	4.50
11.78		4.60		4.50	
11.62	16	4.54	4.56	4.63	4.63
11.68		4.61		4.63	
Average..11.70					
11.81	24	4.67	4.69	4.69	4.64
11.88		4.71		4.59	
11.82	32	4.71	4.72	4.75	4.76
11.78		4.72		4.76	
11.78	48	4.71	4.73	4.79	4.80
11.84		4.75		4.81	
11.88	64	4.73	4.75	4.77	4.80
11.82		4.76		4.83	
Average..11.80					

Table No. 7, though constructed to show the influence of the time of extraction on the amount of extract obtained, is also interesting in this place, moisture determinations having previously been made as required by the official method, and showing a rather interesting variation in this connection.

The data here recorded were obtained on a sample the seal of which had been broken on March 16, being a check sample corresponding to the Michigan Experiment Station, the control analysis performed on that date showing 11.48 per cent of moisture. The bottle containing the sample was then secured with an ordinary cork stopper and stood until May 13, when the first set of eight determinations recorded on table 7 was made. May 13 was a clear dry day, and the analyses showed an average of 11.70 per cent, a gain of .22 per cent over the original sample. On May 14, which was a very damp day, the second set of eight determinations was made, showing on an average 11.80 per cent of moisture, a further gain of .1 per cent. These figures, I think, show the marked influence of atmospheric conditions on the hygroscopic moisture in substances of this nature, and the importance of excluding contact with the air as much as possible.

THE ASH.

Connecticut.—Ash was determined by igniting below red heat till no carbon was left; therefore they could be weighed directly without leaching and evaporation.

Michigan.—Combustion was carried on in a platinum dish, first in an iron bath till substance was charred, then washing with distilled water, and completing combustion in the same dish under a sheet-iron cone to confine and regulate heat. Crude ash obtained in this way was weighed, and pure ash determined by Schroetter's ap-

paratus to eliminate CO_2. The following table shows the difference reported by the Michigan Experiment Station in these two sets of experiments:

TABLE No. 8.

[R. C. Kedzie.]

	Ship stuff.	Corn meal.
	Per cent.	*Per cent.*
Pure ash eliminating CO_2	5.15	.98
Ash by official method	5.40	1.49

South Dakota.—The portions in which moisture had been determined were used for ash.

Kansas.—Ash was determined in same samples as moisture by incineration in a good sized platinum dish.

U. S. Department of Agriculture.—Parallel with the ash determinations by the official method a set of incinerations was made by simply burning to whiteness at the lowest possible heat. For comparison, the results by these two methods are collected in table 9.

TABLE No. 9.—*Comparison of ash determination.*

[A. E. KNOTT.]

By official method.		By ignition to whiteness.	
Ship stuff.	Corn meal.	Ship stuff.	Corn meal.
Per cent.	*Per cent.*	*Per cent.*	*Per cent.*
5.06	1.33	5.08	1.31
5.11	1.35	5.09	1.31
5.14	1.35	5.11	1.31
5.19	1.37	5.15	1.
5.19	1.33	5.15	1.35
5.20	1.41	5.20	1.37
5.23	1.41	5.20	1.29
5.24	1.44	5.25	1.39
5.29	1.45	5.26	1.40
5.38	1.45	5.28	1.40
5.40	1.46	5.29	1.42
5.48	1.50	5.14	1.43
5.49	1.51	5.31	1.46
5.53	1.53	5.35	1.48
5.56	1.53	5.41	1.49
5.82	1.58	5.42	1.51
5.88	1.61	5.54	1.53
Average.. 5.30	1.45	5.25	1.40

It will be observed that the difference on an average amounts only to 0.05 per cent, both for the ship stuff and the corn meal. From these figures it appears that the official method is unnecessarily complicated without an appreciable gain in accuracy. In the opinion of the reporter it is even more liable to error, owing to the additional manipulations required, whereas on the other hand a considerable saving of time would be effected by adopting simple ignition.

ETHER EXTRACT.

Wisconsin.—Following is an extract of a letter from Dr. Woll, of April 17, received with his report:

"As I will be in Europe by the time your association will meet next summer, I take this opportunity to give some data as regards the length of time required for the complete extraction of fodders with ether. The time specified in the directions is sixteen hours. It seemed plain to me that other matters than fat would be dissolved during this prolonged extraction. To investigate the matter, three samples of fodders were extracted for three hours; the extract dried in hydrogen gas and weighed; the flasks were then again connected with the extractors, and the fodders extracted for thirteen hours more. The same process was continued up to sixty hours; the results were as given below:

TABLE No. 10.—*Per cent of ether extract in fodders.*

	Sample 1; ship stuff.	Sample 2; corn meal.	Sample 3; cotton-seed meal.
	Per cent	*Per cent.*	*Per cent.*
After 3 hours	4.55	4.24	9.27
After 16 hours	4.87	4.48	9.47
After 24 hours	4.89	4.52	9.68
After 30 hours	5.02	4.58	9.75
After 40 hours	5.07	4.59	9.84
After 50 hours	5.11	4.59	9.94
After 60 hours	5.11	4.63	9.99

"Each analysis was made in duplicate. It will be noticed that the ether extract constantly increases, even after the sixteenth hour. Sample 1 gained in all 0.24 per cent; sample 2, 0.15 per cent; sample 3, 0.52 per cent. To the percentages after the sixtieth hour's extraction ought also to be added the decrease in the weight of the flasks, due to the action of the water on the glass, three-fourths of which must have taken place after the sixteenth hour. This loss amounted to .0008, .0022, and .0028 gr., on the average for samples 1, 2, and 3, or as 2 grs. of fodder were taken, 0.04, 0.11, and 0.14 per cent, respectively.

"In view of the continuous percentage increase of the ether extract, even after sixteen hours extraction, it would seem that choosing that particular time is wholly arbitrary. Twenty-four, thirty, sixty hours, or in fact any length of time, might as well be taken. In case of the cotton-seed meal there was an unquestionable gain of 0.15 per cent between the fortieth and sixtieth hour of extraction. Some experiments were made with six feeding stuffs, mostly refuse feeds from buckwheat mills, extracting for three, six, ten, and sixteen hours, and drying and weighing as before.

TABLE No. 11.—*Ether extract in fodders.*

	Buckwheat.					
	Bran.	Hulls.	Bran.	Shorts.	Shorts.	Middlings.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
After 3 hours	4.18	.54	1.92	6.42	7.10	8.62
After 6 hours	4.58	.67	2.04	7.65	7.27	8.74
After 10 hours	4.59	.69	2.07	7.76	7.46	8.86
After 16 hours	4.59	.69	2.13	7.79	7.53	8.93

"The increase in ether extract is different in the different fodders, and largest where the highest percentage of ether extract is found. Comparing this table with

the preceding one, there is little doubt but what the percentages given in the last table would have been increased in a similar way after twenty-four, thirty, and forty hours', etc., extraction.

"The question naturally arises, is ether extract to mean fat, or is it to include chlorophyll, organic acids, glucosides, alkaloids, and resins, or other substances but slightly soluble in ether, any one of which might be found in the ether extract after a prolonged extraction. I am not aware how many of them would be removed by the animal charcoal method. I regret that other work did not allow a comparison to be made between this latter method and the ordinary ether extraction method."

The ether used in the determinations was Eimer and Amend's absolute ether, specific gravity .719 at 57.5° C.

New Jersey.—Ether extract was also dried in air, giving practically the same result as by drying in hydrogen, as shown by the following figures reported:

TABLE NO. 12.—*Comparison of ether extract dried.*

[L. A. Vorhees.]

In hydrogen.		In air.	
Ship stuff.	Corn meal.	Ship stuff.	Corn meal.
Per cent.	*Per cent.*	*Per cent.*	*Per cent.*
4.91	4.10	4.95	4.12

Illinois.—Ether extract was also determined on air-dry sample without previous drying in hydrogen, yielding higher results by almost 1 per cent. Following are the figures reported by Mr. Farrington:

TABLE NO. 13.—*Comparison of ether extract on substance not previously dried in hydrogen.*

	Substance dried in hydrogen.		Substance not dried in hydrogen.	
	No. 1.	No. 2.	No. 1.	No 2.
	Per ct.	*Per ct.*	*Per ct.*	*Per ct.*
E. H. Farrington ..	4.68	4.05	5.52	4.72
L. L. Van Slyke	4.88	4.22	4.97	4.26

South Dakota.—A Soxhlet's syphon extractor was employed, requiring 2 ounces of ether and syphoning every fifteen minutes; thus, in the sixteen hours, 128 ounces of ether passed through the substance. In other work it has been customary to continue extraction for forty hours. Drying was done in a current of dry hydrogen to constant weight, requiring eleven and one half hours, using the same apparatus as for moisture. The blackening of the sulphuric acid was again observed.

Kansas.—Ether extract was made with Trommsdorff's absolute ether freshly redistilled from P_2O_5. The extraction apparatus used is constructed wholly of glass and is a syphoning apparatus, the upper ends of the condensers being closed with chloride of calcium tubes.

New York (Genera).—Ether extracts were also made in substance not previously dried in hydrogen (see Table No. 13); and, contrary to the experience at the Illinois Station, no appreciable difference was observed in the amounts of extract obtained from the dried and the undried sample.

New York (Cornell).—Determinations of fat were also made by difference (loss of weight of tubes containing substance) after previous drying to constant weight. (See Table 15.)

U. S. Department of Agriculture.—In the Department laboratory a number of parallel extractions was made extracting with Squibb's stronger ether on the one hand, and with alcohol free ether shaken up with water on the other hand. The results are given in table 14, and as compared with the official determinations by absolute ether it will be seen that the alcohol and water always found in Squibb's ether, and the moisture of the aqueous ether, have an additional solvent action sensibly increasing the extract.

TABLE NO. 14.

Aqueous alcohol free ether.		Squibbs ether.		Absolute ether.	
No. 1.	No. 2.	No. 1.	No. 2.	No. 1.	No. 2.
Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
5.28	4.46	5.42	4.77	4.71	4.03

In addition to the fat determinations by the official method, that is to say by *direct weighing* of the extract in the extraction flask, a complete set of estimations by *difference* was also made in all the check analyses, weighing the extraction tube containing the sample before and after extraction.

TABLE NO. 15.—*Comparison of ether extract.*

	Ship stuff.		Corn meal.	
	By weight.	By difference.	By weight.	By difference.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*
A. E. Knorr	4.61	5.01	3.96	4.20
	4.73	4.64	4.07	4.03
	4.64	4.75	4.01	4.14
	4.71	4.96	4.13	4.23
	4.82	4.92	4.06	4.25
	4.67	4.62	3.91	3.84
	4.67	4.72	4.02	3.98
	4.74	4.65	4.04	*3.48
	4.65	4.69	3.93	3.95
	4.83	4.78	4.03	4.07
	4.76	4.91	3.99	4.11
	4.78	4.92	4.12	4.30
	4.82	4.92	4.01	4.27
Average..	4.73	4.81	4.02	4.12
H. Snyder	4.31	4.37	3.92	3.92
	4.72	4.67	4.04	4.04
	4.56	4.46	.....	.....
Average..	4.53	4.50	3.98	3.98

*Omitted in the above average.

It need hardly be mentioned that in this method the tube must again be dried before making the final weighing, and in doing this in my work I adopted the same *modus operandi* as in estimating the original moisture, drying in a current of dry hydrogen for four hours, though the precaution of drying in an indifferent gas appears hardly to be necessary, since the ether has removed the principal constituents liable to oxidation at a boiling temperature. The figures entered in Table 15 represent the averages of duplicate or triplicate determinations, and show a comparatively insignificant difference between the two sets of analyses, amounting on an average to not over .10 per cent in favor of the determination by difference. By estimating the fat by difference a considerable amount of time is saved, as the determination is made in the same tube and on the same portion of the substance in which the moisture had been previously determined. The tubes require to be dried before weighing after extraction with ether, and this may be done in the same apparatus used for estimation of moisture in the original substance. At least this can be done with the apparatus as arranged in the laboratory of this Department, and which has given such entire satisfaction during the last two years that it has been in use. The fat having been extracted and weighed, the estimation of crude fiber may be further proceeded with on the same portion of the sample, so that operating in this manner, three distinct constituents are estimated weighing out a single portion of sample, and with only a single transfer of substance, from the extraction tube to the digestion flask when the crude fiber is to be digested.

Early in the season some trouble was experienced in getting duplicates in moisture and also fat determinations by weight to agree. This was very soon traced to the sulphurous acid formed in the drying bulbs by the volatile organic substances carried over into the sulphuric acid, and causing a reduction of the same. The odor of sulphurous acid was very marked and the sulphuric acid had turned quite black after four or five determinations. By interposing a U tube with soda lime the sulphurous acid was absorbed, and no further trouble was encountered. Apart from this addition, the arrangement of the drying apparatus as illustrated in Bulletin No. 28 has remained unchanged.

As regards the length of time required for complete extraction, a set of analyses was made on the sample of ship stuff, ranging from four to sixty-four hours, the substance having been previously dried in hydrogen, according to the official method; parallel determinations were made by difference also.

The results of these analyses are exhibited in Table 7. There is a steady increase of material extracted, but by the thirty-second hour this increase becomes negligible, amounting to less than the probable error of analysis. The extreme difference between the extracts at four and sixty-four hours is 0.32 per cent for the series by weight, and 0.46 for the series of determinations by difference, the variation being again in favor of the estimation by difference. It is believed that the higher results found throughout in the latter determinations is either due to small amounts of volatile substances still remaining after the first moisture determination, and driven off during the second drying of four hours, or it may be residual moisture left after the first four hours' drying. Your reporter is not prepared to say which is the case, as no experiments were made by him to ascertain the length of time required for complete expulsion of moisture. In the experience of the New York Experiment Station, at Geneva, as shown in Table 6, this would require from four hours to five hours and a half, making a difference of 0.2 per cent. to 0.4 per cent respectively in the samples sent out this season.

PATTERSON'S CHARCOAL PURIFICATION METHOD.

Patterson's method of purifying the ether extract by percolation through bone-black was tried by four analysts. Mr. Huston, of the Indiana Experiment Station, states that he was unable to obtain duplicates agreeing very closely by using this method, but hardly considers his experience a fair trial. He also tried it on a number of other substances, and, with the exception of the locust bean, which con-

tained only 0.50 per cent of ether extract, there was a marked reduction in the amount and color of the extract. Table 16 gives the results obtained on the charcoal purification method as compared with the figures obtained by the same analysts by the official method.

TABLE NO. 16.—*Comparison of Patterson's method of ether extraction with the official method.*

	Patterson.		Huston.		Parsons.		Knorr.	
	No. 1.	No. 2.	No. 1.	No. 2.	No. 1.	No. 2.	No. 1.	No. 2.
	Per cent.	*Per cent*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
Patterson's method...	4.83	4.00	2.58	2.32	3.98	3.25	3.94	2.94
Official method.	5.53	4.58	4.51	4.02	4.84	4.13	4.71	4.03

THE FIBER.

Wisconsin.—Following is a passage from a letter of Dr. Woll of April 17:

"In the determination of *crude fiber*, after boiling with the acid solution, the filtration was made by means of a small-sized funnel of finest copper gauze (diameter about 1½ inches) fitting tightly in a Bunsen funnel. The solution was shaken before being poured on the filter, and filtered into a clean 500 cc. Erlenmeyer flask; the filtrate was turbid at first, but after a while perfectly clear. The funnel was then placed in another Erlenmeyer flask, and the first filtrate poured back on the filter, and thus a perfectly clear filtrate being secured. The advantages of this method of filtration are: (1) Rapidity of filtration through the loose layer of fodder and asbestos on the copper funnel; (2) the ease of washing the funnel clean afterwards with the hot alkali solution. In boiling the fodder with either acid or alkali solution, a glass tube, 36 inches long, five-sixteenths of an inch diameter, was used as a reflux condenser, and no provision was made to prevent bumping, as there was none; by adjusting the flame properly any danger from this source may be avoided."

The method proposed by Dr. Woll undoubtedly secures rapid filtration, but, in the opinion of the reporter, it would be apt to lead to a somewhat high result, owing to the precipitation of albuminous and other substances, which always takes place on cooling of the solution, it being a common experience in the estimation of crude fiber that both the acid and the alkaline digestion fluids, though they may filter perfectly clear when hot, become turbid and precipitate more or less copiously according to the substance under treatment, when the temperature of the solution falls. In fact the trouble experienced in filtering crude fiber is probably due to this precipitation taking place on the filter, thus occluding the pores, and I believe that this is the principal cause of the differences reported on crude fiber this year and in former years. The slower the filtration takes place, the higher the amount of fiber found will be, this precipitation having taken place in the solution before filtering, the latter in fact in many cases having become quite cold before the last portions of it could be poured on to the filter. In my own experience I find that the more highly nitrogenized substances precipitate more freely than the more starchy ones. By referring to the tables of general averages it will be seen, in support of the above, that Dr. Woll's figures on fiber are considerably above the average, and it is believed that his high results are thus accounted for. In a personal conversation with Dr. Woll on this subject in the early part of the summer, he admitted the possibility of such a precipitation taking place, though he maintained that in his experience filtration was so rapid that the solution did not cool very considerably before having passed through the filter for the second time. At any rate Mr. Woll's scheme is worthy of trial on substances that cause such frequent trouble as cottonseed meal or

linseed meal. In fact this was the only method by which I could secure reasonably rapid filtration in the sample sent out by Dr. Woods, the reporter on highly nitrogenized cattle foods, and the results reported to him from this laboratory were obtained in this way, only using silk bolting cloth in place of the fine wire gauze.

Connecticut (New Haven).—In determining fiber the inverted condenser was not used, because in the methods there employed the loss by evaporation is almost inappreciable; moreover it was found almost impossible to filter the crude fiber on a Gooch crucible. After loss of several determinations in that way, filtering on paper was returned to.

South Dakota.—Fiber was determined in the sample in which ether extract had been made; first filtration through linen, second filtration through asbestos and glass wool. The filtrations were rapid, requiring about fifteen minutes for the linen, and about 12 minutes for the asbestos in Gooch crucibles.

Kansas.—Crude fiber was determined in the residue from ether extract; filtering of the acid solution was done through linen, and it was found an advantage to charge it with asbestos pulp sufficient to fill the bottom of the filter about one-half inch.

Indiana.—In fiber work, silk and linen filters were used side by side. In the acid filtration the silk filtered more promptly, but in the alkaline filtration the reverse is true.

U. S. Department of Agriculture.—In this laboratory both acid and alkali were filtered through asbestos in Gooch crucibles, and, as a rule, no trouble was encountered; but occasionally, for no apparent reason, a filtration would clog and refuse to run. I get the most satisfactory filtration on a Gooch crucible with very large holes; a thin welt of very porous asbestos is formed on this by suspending a very fine fibrous asbestos in ether-alcohol, a small quantity of which is poured into the crucible and allowed to settle by its own gravity, and then igniting. In fact, I prepare my Gooch crucibles in this manner for all of my quantitative work. It gives a very thin film almost resembling the finest filter paper of Schleicher and Schüll, filters rapidly, and will hold the finest precipitates.

THE NITROGEN.

Wisconsin.—In the determinations of protein no zinc was used; otherwise as directed.

New Hampshire.—The official method was followed, except that on albuminoid nitrogen in the corn meal no alum was used. The corn meal contained free acid and gave some trouble in determination of albuminoid nitrogen, as also did the ship stuff.

Kansas.—Nitrogen determined by the Kjeldahl method as by the official directions, except that the arrangement of Armsby (American Chemical Journal, 8, 323) was used to prevent the passage of fixed alkali into the distillate, a current of steam passing through the liquid to prevent bumping and hasten distillation.

New York (Cornell).—Albumenoid nitrogen is reported as made by the official method, and also by adding 20 cc. of K_2S to precipitate all of the copper as well as mercury, which latter would otherwise not all be precipitated, results being slightly higher by this method.

New York (Geneva).—Mr. Van Slyke also reports determinations by Gunning's modified Kjeldahl process, using K_2SO_4 and H_2SO_4 for oxidation, by which he finds 0.2 per cent and 0.1 per cent lower for both the ship stuff and the corn meal.

Dr. Caldwell, in an opinion expressed in one of the former meetings of this association, states that the difference between the highest and lowest results obtained by a number of analysts, on any one constituent, should not exceed 15 per cent of the average, and 10 per cent should be the limit for the widest variation from the average.

Except in case of the fiber and ash determinations in the corn meal, these limits have not been exceeded in this year's work. But, inasmuch as the percentage of

these two constituents is so small, that even a reasonable error would cause a very large variation in percentage of the total amount present, it is hardly fair to judge the results on these constituents on the same basis.

RECOMMENDATIONS.

As to the recommendations for the ensuing year, it depends whether, in the light of this year's experience, we are to continue our methods on the lines hitherto followed, or whether the time has come to widen the scope of, or make radical changes in, these methods for the valuation of cattle foods. In the latter case your reporter has no suggestions to offer, since his entire attention has been directed to a thorough test of the methods and manipulations now in vogue; if, on the other hand, any or all of the present methods for estimating the various constituents are to be retained, a few suggestions as to changes in the minor detail of operation are offered.

First of all, might it not lead to more uniformity of results if some special set of standard apparatus were adopted, especially for the determination of moisture and ether extract, the success of which operations depends so greatly on the strict observance of identical conditions?

For the estimation of ether extract it is recommended that the determination by difference be added as an alternate method, to be used by those whose apparatus for moisture and fat determination can be readily thus applied.

In the estimation of crude ash it would be a considerable saving of time, and an avoidance of manipulations liable to introduce errors, if the leaching with water were omitted; hence it is recommended that incineration to whiteness at the lowest possible temperature be adopted, or, as Dr. Woods suggested to me, incineration alone, if a white ash can be obtained, and if this is impossible, leaching with water as heretofore.

ABSTRACTS OF THE LITERATURE ON METHODS OF FEED STUFF ANALYSIS—JULY, 1890–JULY, 1891.

Adolf Mayer. Valuation of hays by chemical analysis (Journ. für Landw., 39, 107).

The author is of the opinion that chemical analysis, according to the present methods, while giving valuable information on the composition of coarse fodders, still is scarcely adequate for the estimation of their true nutritive value, owing to the great complexity of conditions and circumstances involved.

A given variety of grass, for example, under various conditions of growth and culture, may exhibit greater variations of composition than different varieties of hay when taken under identical conditions.

The digestibility of fodders is subject to great variations. The author also finds that the crude fiber will vary within wide limits according to the condition of comminution.

Proceedings of the Third Meeting of the Association of German Agricultural Experiment Stations. (Landw. Vers.-Stat., 38, 295.)

In these proceedings there is an interesting discussion on the examination of feeds and fodders.

Proceedings at the Meeting of the Committee for Feed Stuffs at Bernburg, July, 1890. (Landw. Vers.-Stat., 38, 141.)

Alcohol-free, absolute ether is adopted for fat extraction. It is the opinion of those assembled that uniformity of apparatus, time of extraction and drying, etc., is desirable, but that definite regulations in that regard are as yet impracticable, and that it only be required for the present to make the extraction complete. The weighed ether extract should be completely soluble in absolute ether without residue. Moisture and fat in linseed-oil cake are to be determined by complete desiccation in an indifferent atmosphere. It is also deemed desirable to determine the acidity of the ether extract, as valuable conclusions may be drawn therefrom as to the age of

the fodder. The importance of the ash determination is also dwelt upon, since feed stuffs sometimes are found to contain considerable quantities of sand.

Walter Maxwell. Methods of Estimation of Fatty Acids in Vegetable Substances. (Amer. Chem. Journ., 13, 13–16.)

From experiments on cotton seed and legumes, the author concludes that the substituted glycerides (lecithines) are but imperfectly extracted by the ordinary methods of ether extraction as practiced in feed analysis.

J. König. Examination of Acid and Compressed Fodders, and the Estimation of Total Nitrogen in them. (Landw. Vers.-Stat., 38, 227–234). This article consists largely of a compilation of analyses.

O. Förster. Apparatus for Drying the Fat Extract in Feed Analysis. (Landw. Vers.-Stat., 37, 57).

The author gives drawings and a detailed description of an apparatus for drying in an atmosphere of carbonic acid or illuminating gas.

G. Loges and C. Claessen. Estimation of Free Fatty Acids in Fodders. (Landw. Vers.-Stat., 38, 314–316).

The authors make comparison of four methods for estimation of fatty acids in fodders:

(1) To extract with ether and titrate after previous drying for three hours.

(2) After three hours drying to add alcohol to the ether extract and then titrate.

(3) Titration without previous drying.

(4) Digestion for three hours in cold ether, and titration of aliquot portions of the filtrate after addition of alcohol.

The authors find the values for 1 and 2 too low, owing to volatilization during drying, and hence recommend either 3 or 4.

Z. v. Milkowski. Estimation of Starch in Grains. (Zeitschr. Anal. Chem., 29, 134–136.)

The author has made numerous comparative estimations of starch in barley by the methods of Maercker and of Asboth, and concludes, from the agreement of his results, that both methods are entitled to confidence.

A. Leclerc. Estimation of Starch in Fodder. (Journ. de Pharm., 21, 641–645).

Weigh off 2 gr. of powdered grain, or 5 gr. of straw, hay, etc.; place in a 250 cc. flask with 10 cc. of water. Add 180 cc. of a solution of zinc chloride, sp. gr. 1,450, and heat in a salt-water bath for 1 to 1½ hours at 105°. Cool and make volume to the mark. Filter and to 25 cc. of the solution add 2 cc. hydrochloric acid and then 75 cc. 90 per cent alcohol. After twenty-four hours, filter through a tared filter. The whole of the dextrin and starch are precipitated, sugar remaining in solution. The precipitate is washed with 90 per cent alcohol containing one-half per cent hydrochloric acid, and finally with alcohol alone. The mineral matter carried down by the starch is deducted after incineration. The formation of a little dextrin may be disregarded, having the same composition as starch.

O. Reinke. Estimation of Starch. (Zeitschr. Anal. Chem., 29, 472–475).

The author considers two distinct processes for the estimation of starch: with the use of pressure, and without pressure, and recommends the following as the best:

With pressure.—3 gr. of the finely powdered substance are stirred with 25 cc. of a 1 per cent solution of lactic acid and 30 cc. of water in a metallic beaker, then covered and heated for two and one-half hours in a digester at 3 atmospheres pressure, then mixed with 50 cc. of hot water, and after cooling made up to 250 cc. and filtered. 200 cc. are then inverted with 15 cc. of hydrochloric acid (1.125 sp. gr.), neutralized with soda, made to 500 cc., and 25 cc. titrated with Fehling's solution.

Without pressure.—3 gr. of the substance are boiled with 50 cc. of water and then digested for an hour at 62.5° C. with 0.05 gr. of diastase, cooled, made to volume of 250 cc., and 200 cc. inverted as above. Detailed directions for preparing the diastase are given.

REPORT OF COMMITTEE ON ESTIMATION OF FIBER IN SUBSTANCES RICH IN CARBOHYDRATES.

[See Proceedings, p. 75.]

The two reporters on cattle foods recommend for the estimation of fiber in substances rich in carbohydrates other than fiber, the use of 2.5 per cent acid and 2.5 per cent alkali solutions in place of the strength of 1.25 per cent, as required by the present method for cattle foods low in carbohydrates.

In all other particulars the general directions given for the analysis of cattle foods are to be followed.

It is further recommended that the reporter on starchy cattle foods appointed for the ensuing year test the method of Hönig (report A. O. A. C., 1890, p. 81), for the determination of fiber and starch, and request the coöperators to make comparative tests on the same.

Thus the official directions would be changed to read as follows:

(*a*) *For feeding stuffs poor in starch.*—Extract 2 gr., etc.

(*b*) *For feeding stuffs rich in starch.*—Same as method (*a*), substituting 2.5 per cent acid and alkali solutions in place of 1.25 per cent solutions.

AUGUSTUS E. KNORR.
CHARLES D. WOODS.

Mr. WOODS (after reading the report of committee). Our recommendations would be that on p. 212 of the report, A. O. A. C., 1890, in the method of analysis, which is now modified to be the official method for the analysis of cattle foods irrespective of whether they are starchy or nonstarchy, under crude fiber, the method given be described as (*a*) "Feeding Stuffs Poor in Starch," and this be added: (*b*) "Feeding Stuffs Rich in Starch—same as method (*a*), substituting 2.5 per cent acid and alkali solutions in place of 1.25 per cent solutions."

Mr. WITHERS. What are some of the reasons for the change?

Mr. WOODS. The chief reason is the difficulty of handling starchy feeding stuffs with as weak an acid as 1.25 per cent, the starch being more readily handled with the use of the stronger solution. It removes all danger arising from imperfect filtration, or very nearly so, and makes very little difference in the percentages of the fiber.

Mr. KNORR. In addition to what Mr. Woods has said, I refer to my results in the tables of Dr. Caldwell's report of the previous year on wheat middlings, on which 5 per cent acid had been used instead of 1.25 per cent, as required by the official method, the results being identical with the average of all the analyses using 1.25 per cent, and the manipulation of the fiber being greatly facilitated by this method.

Mr. JENKINS. In what respect is manipulation easier?

Mr. KNORR. The filtration is very much easier and more rapid.

Mr. HUSTON. Would there be any objection to making the alkali stronger in determinations of material not containing much starch? I have been convinced that there is nitrogen still remaining after the fiber determinations, if the methods are closely followed. I do not want to make a motion to change the method, but think it would be well to recommend that experiments be made upon this class of material as to the strength of the alkali, and nitrogen determinations be made in the

resulting fiber. My experience has been that a fiber which did not filter well after treatment with alkali was one in which nitrogen still remained.

Mr. WITHERS. I have found that the strength of the acid, as well as of the alkali, makes a difference in the amount of crude fiber, and I think 2.5 per cent acid and 2.5 per cent alkali ought to be used in both cases, so that the method may be uniform; otherwise the crude fiber will not correspond.

Mr. COOKE. I move that the report of the committee be accepted and adopted. Carried.

Mr. JENKINS. I have a couple of papers which I had intended to present at the meeting of the Chemical Section of the Association of American Agricultural Colleges and Experiment Stations, but as those sessions have been merged with those of this association, I will simply refer to them. They are a further report of results which are briefly alluded to in a paper presented to the association at Champaign in regard to the investigation or reinvestigation of the albuminoids of some of our seeds, grains, cereals, etc., with the hope of getting more light on their composition. Dr. Osborne has been engaged for two years in this work, and the results already obtained are interesting and encouraging. He has succeeded in isolating crystalline albuminoids in large quantities, preparing them by different methods, and by fractional preparation, and finding that the chemical composition of the various fractions was identical. Dr. Osborne's work also shows that the factor 6.25 is not very far from correct.

Mr. WITHERS. What kind of filter does the reporter on foods poor in carbohydrates use in determining crude fiber? Is the filtrate clear, and how long does it take to filter?

Mr. WOODS. For the first and second filtrations linen is used. I never have any difficulty in the filtrate passing through, and the filtrations are uniformly rapid, although in such things as cotton seed the first filtration possibly takes six minutes; ordinarily it would take about a minute. After making the second filtration on linen, and washing on the linen, until it is free from alkali, it is transferred to a beaker and filtered on a Gooch crucible in the usual way. The time used in making the first filtration with linen is more than saved by the benefit derived.

Mr. WITHERS. I have found considerable trouble, after treating with sulphuric acid in the determination of the fiber, to get the filtrates clear and the filtration rapid; and, after some experiments, have noticed that by changing the order of treatment I get a fair filtrate, and the filtration takes place in the first case in about three or four minutes, possibly a little longer, and in filtering with the acid the filtration takes place in about a minute. I would like to ask whether any other member who worked on that line had trouble in getting rapid filtration after treating with acid, and whether it was always clear?

Mr. HUSTON. I have tried reversing the order of treatment, especially on linseed meal, and the first time lost five days in the operation,

and did not continue the experiment. I have recently made some experiments in filtering on asbestos and linen and silk, and the best result obtained was with heavy white silk, with which, however, it is necessary to exercise care in drying, it being best done by pressure; it can not be wrung out as in the case of linen.

In regard to fiber, I am sorry that no one has determined the nitrogen left in it. In working on fiber recently I found 1 per cent of nitrogen in what purported to be crude fiber.

Mr. SHEPARD. I desire to call attention to the necessity of care in packing the tube in which the samples are placed for ether extract. I closed the bottom of the tube with linen of various grades, and made a great many determinations. Afterward, in endeavoring to dissolve the ether extract obtained, I found there was an insoluble residue, especially in the case of natural grasses, and have come to the conclusion that the filtration is not close enough, and that these were fine particles which had been ground up, or perhaps fine particles of dust which ultimately turned up as ether extract. I then used No. 589 S. & S. filter paper and obtained perfect filtration, and found that the ether extract immediately became considerably lower, and also perfectly soluble.

Mr. HARRINGTON. Were the extractions made on green grass or grass that had been dried?

Mr. SHEPARD. The grass had been hanging in the laboratory until thoroughly air-dried, very little coloring matter being left.

Mr. HARRINGTON. I inquired on that point particularly, because I am satisfied that in my own work with green grasses the ether extract was far higher than it should be in many cases. I have endeavored with various substances to get something that would extract the fat without this trouble, but have been unsuccessful. I have met with the same trouble mentioned by the last speaker in redissolving the extract in ether, and had remaining behind chlorophyll that was unmistakable, and in many instances other coloring matter. I have got coloring matter from the seed of sorghum, and am satisfied that in certain instances ether would remove coloring matter in considerable quantity that would count for fat, when really it was not fat at all.

Mr. KNORR. This coloring matter can be removed by Dr. Patterson's method.

Mr. PATTERSON. I have found no difficulty whatever in getting rid of the coloring matter by the charcoal method described to the association a year previous. The fat would have a very different appearance, and in most cases be entirely soluble again, and looked just like pure oil or fat.

Mr. SHEPARD. Would not some of the oil be oxidized in filtering through charcoal?

Mr. PATTERSON. With pure fats there is no change whatever. I have used pure fats with sorghum and cotton seed, and the weights were practically the same after passing through charcoal as before.

Mr. FREAR. I desire to call the attention of the association to some results obtained by Mr. Fries, one of my assistants, in making ash determinations of wheat; a very difficult determination to make. Even when burned at the lowest temperature possible, and repeatedly extracted with water, it had a constant tendency to fuse and adhere to the sides of the dish, requiring a good deal of care to remove it thoroughly. Mr. Fries had been led to try very finely powdered quartz, and in that way had been able to heat his sample at a moderate temperature, and it became quite red without any fusion whatever, very satisfactory results being obtained. I think this method worthy of trial in cases where difficulty is experienced on account of the presence of very fusible salts in large quantity.

Mr. SHEPARD. I have hastened the time of burning to whiteness in ash determinations by the addition of ammonium nitrate, and would like to know whether there is any objection to such addition. Fresenius recommends it, and I have tried it and obtained exact results.

Mr. VAN SLYKE. I have made several experiments, using nitric acid instead of ammonium nitrate, and expect during the coming year to make many more. My experience indicates that there is practically no difference in the results.

Mr. HARRINGTON. I have not had experience with ammonium nitrate, but have done work with nitric acid and sulphuric acid by the official method, burning off in a muffle over a gas furnace. My work shows that the results would depend upon the character of the material in which the ash was determined, being pretty nearly concordant with some materials, and higher with others. Whether or not ammonium nitrate would have the same influence, I can not say.

Mr. KNORR. With most materials there is no difficulty in obtaining a perfectly white ash if the incineration be carried on slowly enough, and at low enough temperature, but if the temperature is allowed to rise too high it causes the ash to fuse and occlude the particles of carbon, thus preventing the oxygen of the air from oxidizing them. I burn my ashes at the lowest possible heat in a muffle.

Mr. ROSS. I have tried the method suggested by Mr. Frear, not with regard to determining the per cent of the ash, but preparatory to determining the potash in cotton-seed meal; have found that by mixing with silica, a very nice ash could be obtained. Of course, as has been shown, there was loss of potash, but the experiments were made merely to see whether or not an ash could be secured with very little trouble with regard to charring, and the results, at least in that particular, were quite a success. One or two experiments were made with ammonium nitrate also, but the results were not so satisfactory as by the method I have just referred to.

Mr. JENKINS. Matters will be facilitated by taking up section by section the official methods of the previous year for discussion, and to that end I move that the first section of the official method for the analysis of foods be adopted by the association for the coming year. Carried.

Mr. WOODS. I desire to call attention to the title of the methods for the analysis of foods as given in the proceedings of the previous year, and ask whether that title is to be retained, referring, as it does, only to nonstarchy foods? If so, should the association have a new method that would cover starchy foods, or make the methods given include feeding stuffs, irrespective of whether they contain starch, oil, or nitrogen?

Mr. JENKINS. I move that a single method be adopted for the analysis of all foods and feeding stuffs for the ensuing year.

Mr. WOODS. Unless I am mistaken the change of titles last year was made without any action of the association.

Mr. KNORR. The matter was simply overlooked. The division of the work having been made, and these methods having been laid down for nonstarchy foods, the starchy class was subsequently forgotten.

Mr. SHEPARD. The wording of the title as it stood would imply that there was another method given somewhere, which is not the case. Therefore I am in favor of restoring the original title.

The PRESIDENT. I supposed, when the appointment of reporters was made, that the reporter on starchy foods would propose some method for the analysis of that class of foods.

Mr. KNORR. The reporter did not feel that he could take it upon himself to establish a new method, inasmuch as he had no authority to do so.

The PRESIDENT. The question is then whether the old method should be restored.

Mr. SHEPARD. I do not understand that the change would mean anything more than a return to the original title of the method, making it read, "An official method for the analysis of cattle foods."

Mr. VAN SLYKE. I understand that this would not imply that two subdivisions could not be provided for, but that the work is to be done under two subdivisions until two sets of methods are decided upon.

The motion was then put and carried.

Mr. KNORR. I move the adoption of the second section, with the amendment that an apparatus be recommended in which the hydrogen would pass through the substance instead of only over it. Certainly more moisture will be taken off by the hydrogen passing through the substance than otherwise.

Mr. VAN SLYKE. I have made a special set of experiments, using Prof. Caldwell's apparatus, to see whether I could use one stream of hydrogen, passing the stream of hydrogen through sulphuric acid in order to take up the moisture. I could not make it work, at least as far as I went; it is probable that I did not use sulphuric acid in sufficient quantity.

Mr. KNORR. There is an apparatus at the Department of Agriculture devised by Dr. Wiley and myself, which I would like to invite Mr. Van Slyke to inspect.

Mr. HUSTON. I have experimented with an apparatus similar to Mr.

Knorr's, except that I use solid caustic potash in small lumps in place of his chloride of calcium, and have been very successful. I have thirteen tubes in the series, and have never been able to detect any difference between the first tube and the thirteenth, or anything else to indicate any difference in the drying qualities. Nor have I been able to detect that the marked difference in pressure between those two tubes made any difference in the amount of moisture.

Mr. JENKINS. The amount of water got out of the substance will depend very considerably upon the dryer used to dry the gas before introducing it. Hydrogen could be so dried by oil of vitriol that it would be dry to chloride of calcium; it could be so dried by chloride of calcium that it would be dry to oil of vitriol of certain strengths. If the work was to be absolutely alike, one material would have to be used for drying the hydrogen. The use of chloride of calcium and sodium hydrate has been discontinued at my laboratory for the reason that they grew weaker. A form of apparatus is now used with which the gas is dried by a slow stream of 90 per cent oil of vitriol trickling over glass beads. That oil of vitriol is never used a second time. With that apparatus we have been able to run a very rapid stream of hydrogen, and have never been able to gather any moisture from the hydrogen that has gone through, even with the best sodium hydrate tube or calcium tube that is made.

Mr. WOODS. The more the current of hydrogen is passed through the substance, the more material there will be carried over, and the faster will SO_2 be formed. While apparently higher results of water would be obtained, the increase would not be due to moisture, but to other volatile material. I therefore think it unadvisable to insert the requirement suggested by Mr. Knorr; but it would be a good idea to recommend that the reporter take this matter into consideration and study it in the work of the ensuing year.

Mr. SHEPARD. I agree with Mr. Woods that it is not desirable to lose any more of the volatile matter than can be helped, and think the change would be unadvisable at present.

Mr. KNORR. The difficulty is to draw the line as to where moisture stops going off and other volatile matter begins to escape, and I think that inasmuch as the loss of a portion of the volatile matter can not be avoided, it would be better to make the error a constant one by getting rid of it all.

Mr. TELLER. Have any efforts been made to remove the oxygen that always seems to form in the generation of hydrogen? It seems that a certain amount of oxidation will take place unless it is removed before drying.

Mr. Knorr's motion was then put and lost, and on motion the section was adopted without change.

Mr. SCOVELL. I move that the method for the determination of the ash be amended, so that the amount of the substance to be taken should be stated.

Mr. WOODS. I move to amend the amendment by specifying the amount as 2 to 3 grams.

Mr. MCDONNELL. I do not see the use of taking more than 2 grams. It would make it difficult of incineration.

The amendment of Mr. Woods was put and carried, and Mr. Scovell's amendment as amended was carried.

Mr. KNORR. I move as a further amendment to the method, that the incineration be carried to whiteness, if such can be obtained, at the lowest possible red heat; otherwise that the use of the method of the previous year be recommended. Carried.

Mr. VAN SLYKE. If it be not out of order, I move that the reporters on fodders for next year be requested to take the study of ash into consideration as the result of the remarks that have been made on the subject, treating with nitric acid or ammonium nitrate or quartz, and making comparisons. Then the association will have some data by which to be guided.

The PRESIDENT. I suggest that Mr. Van Slyke bring up that matter later as a recommendation to the reporter.

Mr. WINTON. I desire to call attention to the direction in the method that the substance should be charred at a low red heat, and ask if it is not generally supposed that alkali chlorides go off at a low red heat. I think experiments have shown this to be true, and although inclined to doubt it, I am desirous of knowing what the other members think about it.

The PRESIDENT. I am not acquainted with any investigations that would prove it.

Mr. WINTON. I have always been careful in burning ashes to keep the heat to low redness, and have been able to obtain perfectly white ashes even with very starchy materials like Indian meal or wheat, and without the use of a muffle furnace. There is a muffle furnace at my laboratory, but it is not very well liked. It works very well when a single ash determination is being made, but when it is filled with half a dozen dishes the heat is not properly distributed. By a very simple device we have obviated the necessity of using a muffle. The burning is done in a flat-bottom platinum dish on a very thin piece of asbestos paper. In this way a very high heat can be obtained and equally distributed over the bottom of the dish. In some cases where this proved insufficient, a little piece of the same asbestos paper was laid over the dish in such a way as to cause the heat to be equally distributed.

In some cases, however, where we have not had patience to wait for it to burn to complete whiteness, we have found it more convenient to determine the charcoal in the ash, rather than to attempt to burn it and weight it. The ash is ignited as far as convenient and then weighed; after that, boiled with acid, and the carbon collected on a Gooch crucible and weighed. I thought this much more convenient than other methods.

Mr. PAYNE. I have made a number of experiments in regard to the

volatilization of chloride of potash, especially in fertilizer work, and have found a loss of chloride of potash at low red heat, and a loss of sulphate of potash when heated to whiteness.

Mr. PENNY. I will mention, as of interest, the practice among authorities having come up, that of Bunsen, a rather careful analyst. In his determination of potash, he drives off the water at a very faint red heat, so faint as to be scarcely visible. In his opinion it would not do to remove the lid long enough to seize the crucible with a pair of tongs for fear of volatilization. He requires it to cool to such a point that it can be touched with the hand.

Mr. HUSTON. We have been working from a somewhat different standpoint. We do not care how much chlorine gets away, but do not want any potash to be lost. One of my assistants ignited a large platinum dishful of the material, and then duplicated a good deal of the work, the results having been questioned, barely getting the material charred well, and the results were practically identical. The same man had been igniting sulphate of potash with big cannon-shaped blowpipes, and had not been able to detect any appreciable loss of sulphate of potash, although the ignition had been carried on for some time. All these ignitions were performed in broad open platinum dishes.

The motion to adopt the section as amended was put and carried; and the convention then adjourned until Friday morning at 9:30 o'clock.

SECOND DAY.

MORNING SESSION, FRIDAY.

The convention was called to order by the president at 9:30 o'clock, and the deliberations on the subject of feeding stuffs were resumed, the determination of ether extract being taken up as the next subject in order.

Mr. JENKINS. Is it the general experience that extraction for sixteen hours is necessary with most fodders? I consider it necessary to get perfect extraction, whether it takes one hour or fifty. In my regular laboratory work, having satisfied myself that, with the particular class of fodders under examination at any time, I could get as much in four or five hours' extraction as in sixteen, I do not consider that I am bound to hold to the sixteen hours. I have never had experience with anything that required sixteen hours for extraction. Neither do I dry in hydrogen before beginning the extraction. I prefer to have the wording of this method changed, so that those using it would be directed to perfectly extract with anhydrous and alcohol-free ether.

Mr. WILEY. I desire to call attention to the data mentioned in the report on this subject, which show that the kind of apparatus used makes a great difference. In using a siphoning apparatus the reporter

had suggested that it would be better to designate a certain number of siphonings instead of extraction within any certain time, but with an ordinary percolator the time element should be considered. But while the data showed that between three hours and sixty hours there was quite a difference, the increase after the twelfth or fourteenth hour was extremely slight. If the direction were to extract perfectly, there would be great difference of opinion among analysts as to the meaning of that. If a limit of contact of the extracting material could be fixed, the results would probably be more concordant. Of course it is to be remembered that all ether extract is not fat, although most of it is composed of glycerides soluble in ether. Last year it was pointed out by work done in the Department of Agriculture that certain substituted glycerides could not be dissolved in ether except after a time totally out of the limit of ordinary analytical work, and such substituted glycerides would have to be extracted by a mixture of alcohol and ether. So all the fat was not to be obtained with ether. Many of the lecithin principles would remain in the substance after extraction for twelve or sixteen hours in ether. Of course this referred especially to lupines and other seeds containing large quantities of substituted glycerides. What is wanted is to get a certain constant practice, so that the amount of extract obtained by each analyst would be obtained under practically the same conditions. If a limit of time is set, all would work that way; but if the direction is to be that extraction is to continue until there is no further loss of weight, it should be modified by saying no appreciable loss of weight. If after five or six hours' extraction there is an increase of only one-hundredth per cent, that should be neglected, and the extraction be considered as complete.

The PRESIDENT. This subject should be considered very carefully, as ether extract is the determination with which most trouble has been experienced. I think sixteen hours better, as Mr. Wiley has suggested; and that some time of contact should by fixed upon.

Mr. JENKINS. I only wanted to obtain an expression of opinion on this subject. In analyzing samples sent out for comparative work I, of course, follow the directions precisely, but in my regular routine work, if I could only raise one one-hundredth of 1 per cent after three or four hours' extraction, I do not feel that neglecting that amount is serious enough to justify me in consuming the extra time.

Mr. KNORR. Dr. Woll shows that extraction is approximately complete at the end of about thirty hours, which is corroborated by my own experience; but up to that length of time there seems to be a steady increase, and I do not think it would be safe to reduce the time. The substance experimented upon by myself was ship stuff, containing about 5.71 per cent of fat. Dr. Woll experimented on ship stuff, corn meal, and linseed meal sent out by Dr. Woods, and also on refuse from buckwheat mills—hulls and bran.

The PRESIDENT. Are there any further remarks on the subject of extraction?

Mr. KNORR. I move that the determination of ether extract by difference be adopted as an alternate method.

In justification of this motion I beg leave to call attention to the statement made in my report in regard to this matter, to the effect that all the ether extracts made in my control analyses were at the same time checked by determinations by difference, and, the work having been done in triplicate mostly, these figures represent upward of one hundred analyses, the discrepancies between the two parallel determinations being on an average one-tenth of 1 per cent in favor of the determination by difference, which is within the limit of error; and, inasmuch as a considerable saving of time could be effected in determining the extract by difference, I think it would be an advantage to insert this as an alternate method at least.

The PRESIDENT. I desire to call attention to the paper read by me yesterday, in which it is shown that one of my assistants had obtained results by this method which were practically the same, provided the substance was dried somewhat longer—generally until the loss was not more than 1 mg. in the substance taken.

Mr. VAN SLYKE. In the description of the method the precaution mentioned by Mr. Caldwell should be inserted as a part of the method, namely, that drying be continued until the loss is reduced to 1 milligram.

Mr. KNORR. I accept that as an addition to my motion.

The motion was then put and carried.

Mr. KNORR. I move to amend the section under consideration by adding that some one form of apparatus be adopted for the extraction, in order to make the manipulation as nearly uniform as possible.

Mr. WOODS. Inasmuch as nearly every laboratory is already equipped with some form of ether extraction apparatus, which would not be willingly thrown away, it might be well to adopt Mr. Knorr's motion as a recommendation, but I do not favor it as a requirement.

Mr. FREAR. I suggest that before making recommendations in regard to particular forms of apparatus, it is necessary to have comparative tests made to determine which form of apparatus should be recommended. I do not think it essential to have all analysts use one form of apparatus, as different results would be produced even with the same form of apparatus when the work is done under different conditions. This matter should be one of test rather than of adoption at present.

Mr. JENKINS. I agree with Mr. Frear. If the analyst be careful and capable, he will get correct results with any decent form of apparatus. On the other hand, if he be careless in his work, no form of apparatus he might use would enable him to obtain correct results.

The motion was then put and lost.

Mr. WOODS. I think the words "in a current of dry hydrogen" are unnecessary in this method, and I move that they be struck out.

Mr. DE ROODE. I have made some experiments on this point. I have dried to constant weight in hydrogen, and then, to see if it would have

any effect, afterwards placed in a dry air bath and heated for fifteen or twenty minutes, and in no case did I find any appreciable increase in weight. In some cases I have found a slight loss by drying in air, showing that in these cases, in which hays, grasses, etc., were used, no appreciable oxidation took place.

Mr. SHEPARD. I have found it necessary to dry the ether extract until it became solid. I have found that as long as any portion of it remained liquid, it would continue to lose in a current of dry hydrogen. I have found it necessary in some cases to dry as much as nine hours before the loss would cease.

Mr. DE ROODE. I think it would depend altogether upon the kind of oil used. Waxy substances would get solid, but oil like olive oil would not. I certainly do not think that such bodies should be dried until they are solid.

The PRESIDENT. I think that the methods should be practically accurate, but do not think that time should be sacrificed in order to make them superaccurate. They should be such as can be carried out in practice.

Mr. VAN SLYKE. I have found no appreciable difference between the results obtained by drying in hydrogen, and drying as long as two hours in an air bath, on the samples sent out by the reporters.

Mr. WINTON. I desire to ask Mr. Van Slyke whether he has obtained any comparative results on linseed oil. Some experiments have been published abroad on this point, and I would like to know whether any have been made in this country?

Mr. VAN SLYKE. I know of none.

Mr. JENKINS. I think it perfectly certain that linseed oil could not be properly dried in air. It would oxidize readily, and as soon as it became oxidized, it would be soluble only in chloroform. I consider it necessary with certain classes of fodders to dry in hydrogen.

Mr. WOODS. I do not think there is the slightest question that any vegetable substance, such as corn or linseed, if dried for any continued time in air, would no longer yield up its full amount for extraction; but that does not seem to me to be the question under consideration, which is whether the ten, fifteen, or twenty minutes necessary to dry the fat would so oxidize it as to make a perceptible increase in weight. It does not take more than twenty minutes at the outside at the temperature of boiling water to expel all the ether from the fat flask, and experiments in my laboratory show practically no difference in results.

The motion was then put and carried.

Mr. WOODS. I move that the section on ether extract as amended be adopted.

Mr. KNORR. I think, before proceeding to the final motion, the quality of the anhydrous alcohol-free ether should be more explicitly specified. A lot of absolute ether imported from Germany for use in our laboratory, which had been guarantied to be absolutely anhydrous and

alcohol-free, was tested with metallic sodium, and there was found a copious evolution of hydrogen, showing that it was not absolute ether, as claimed; and all the absolute ether used thereafter in the laboratory was prepared with metallic sodium. Moreover, ether can not be kept anhydrous for any length of time unless kept over metallic sodium. I move to insert the words "specially prepared" before the words "anhydrous and alcohol-free ether." Carried.

The section as amended was then adopted, reading as follows:

Extract 2 to 3 gr. of the substance, dried as for the determination of the moisture, with specially prepared anhydrous and alcohol-free ether, sixteen hours. Dry the extract by exposure to the full heat of boiling water to constant weight.

Mr. VAN SLYKE. I move that the method for crude protein be adopted without alteration. Carried.

Mr. WOODS. I move that the method for albuminoid nitrogen be adopted without alteration.

Mr. FREAR. I think a clause ought to be inserted which would secure the precipitation of the copper in accordance with the results reported by the president, so that there might be no residue of mercury left to interfere with the process.

Mr. DE ROODE. I desire to call attention to the paper read the previous day by the president on the subject under consideration, and move that the process recommended in that paper be adopted.

The PRESIDENT. I will explain that the method referred to simply provides for the complete precipitation of the mercury by adding enough potassium sulphide to precipitate both copper and mercury.

The motion was then put and carried, and the method for albuminoid nitrogen as amended was adopted.

Mr. LUPTON. I move that the method for crude fiber be adopted without alteration. Carried.

Mr. WOODS. I move that the subject of dairy products be reconsidered, in order to take up the methods for the analysis of sour milk recommended by Mr. Cooke. Carried.

Mr. Cooke read the following recommendations of methods for the analysis of sour milk, which were adopted. (See Methods of Analysis, p. 203.)

Mr. Cooke also read a method for making total solids in very sour milk, which was adopted as a part of his report on dairy products, but not included in the official method. (See Cooke's report, p. 22.)

Mr. WILEY. I move that the reporter for the ensuing year be requested to investigate the influence of citric acid in milk on the quantity of fat extract obtained by the usual method. This subject has not received attention at the hands of reporters in previous years, but the matter has recently been investigated abroad, and the amount of citric acid in normal milk determined. It is well known that normal milk contains a large quantity of citric acid, and I think it as important as lactic acid, because of its being a constituent of normal milk in combination with lime salts. Carried.

Mr. VON SCHWEINITZ. Dr. John A. MacWilliams, of Aberdeen University, in an article in the British Medical Journal of April 19, 1891, suggests the use of sulpho-salicylic acid as a test for albumin in urine. As sulpho-salicylic acid would precipitate from solution all classes of proteids, this reagent might be of use in determining soluble albuminoids. These bodies may be divided into the following 6 groups:

(1) Native albumins (egg albumin and serum albumin).
(2) Derived albumins (acid and alkali albumins).
(3) Globulins (serum globulin, myosin, vegetable globulins).
(4) Fibrin held in solution by dilute alkalis or neutral salts.
(5) Protoses (albumoses, etc.)
(6) Peptones.

It was found that sulpho-salicylic acid would precipitate all of them. The precipitate formed upon boiling with an excess of the acid, in case of groups 1 to 4, would become flocculent but not coagulated, while the precipitate with groups 5 and 6 would dissolve and separate out again on cooling. This would give an easy and rapid method of determining the presence of these two classes of proteids in a solution. The test is a very delicate one, and I think would prove very valuable in studying the proteids in general, both qualitatively and quantitatively, and it is susceptible of further development. I suggest that the members of the association engaged in this class of work try this method in their investigations.

Mr. COOKE. It may be supposed from some of the figures presented in my first report that it is not possible to analyze buttermilk accurately by the Babcock whirling method, but this is not the case. I have investigated that question thoroughly, and find that if quite a decided excess of solid sodium hydrate be added to buttermilk, it will bring it back to complete fluidity, and it can be analyzed with accuracy by either the Babcock or Beimling method, and the same is true of sour milk. The alkali should not be added long before the determination is to be made, because in that event saponification would take place; but if the determination be made immediately after adding the alkali, there seems to be no danger of saponification, even with a large excess of the alkali. Soda is better to be used in this determination than ammonia.

Mr. WILEY. I call attention to the fact that the association has adopted no method for the analysis of cereals. This subject was mentioned the previous year, and that was one of the chief reasons for making the division of the work. I think it strange that the association has never adopted a method for the analysis of cereals, as they constitute the most important product of the farm for cattle food. I therefore move that the reporters on feeding stuffs—the present reporters—be requested to present to the meeting before adjournment a scheme for the analysis of cereals and other foods rich in starch. Carried.

Mr. VAN SLYKE. I move that the reporters on feeding stuffs for the ensuing year be instructed to make a study of methods for determining

ash, especially with reference to the use of quartz, nitric acid, and any other substances that they may see fit. Carried.

Mr. WILEY. I suggest leaving out the name of the sodium hydrate company in the method for crude fiber. It was agreed to.

Mr. SHEPARD. I suggest that in next year's work only one subject be assigned to each reporter. For instance, have a reporter on ash determinations, and let him send out to the different stations a large number of samples covering the various kinds of material, so that a better comparative test of methods can be obtained, and so with the other determinations.

The PRESIDENT. This is quite a radical change, and I suggest that Mr. Shepard bear it in mind to bring it up after the consideration of the different reports has been finished, so that it may be made to cover all the subjects.

Mr. WILEY. At the suggestion of the president I will state that an attempt will be made to furnish the reporters for the ensuing year, in advance of the publication, as soon as it can be collated, a typewritten copy of the methods adopted and the recommendations as to alterations. I hope the reporters will remember that the methods which are adopted are to be handed to the secretary of the association with their reports. I am depending on the reporters to make account of all the changes, as I can not read the stenographer's notes until they are transcribed.

Mr. Stubbs presented the following report:

REPORT ON SUGAR ANALYSIS.

By WILLIAM C. STUBBS, PH. D., *Audubon Park, New Orleans, La.*

The able report made by my predecessor, Dr. H. W. Wiley, at your last meeting, and published in the proceedings of the Seventh Annual Convention of the Association of Official Agricultural Chemists, makes easy the task assigned me.

Since our last meeting a large amount of chemical work has been done on sugar and its products. It was intended during the past year to distribute samples of sugar, molasses, candies, and honeys among the members of this association for analysis, but the hygroscopic tendencies of these substances, together with the difficulties still existing, of determining sucrose in the presence of other rotatory bodies, made me skeptical as to the value of the results. Only one station applied for samples, and to it the reply was made that it was too late in the season to collect proper samples. If it be the desire of this association to distribute samples of these products among the members, as is done with other substances, my successor can be so instructed, and I am sure will gladly perform the work.

The increased interest in growing sugar in this country, and the gradual tendency of the times towards extirpating adulteration from our food products, and especially the new national law giving a bounty on sugar made in the United States, varying with the polariscopic test, all serve to render chemical information relative to sugar worthy of a recital in our proceedings. Already the national Department of Agriculture, with its branches in various parts of the country, has done much to enlighten the growers of sugar cane, sorghum, and beets. Sugar-experiment stations have been established in Louisiana, Florida, Kansas, and Nebraska, and earnest work is being accomplished. During this year Drs. Wiley and Maxwell have published

remarkable researches into the amorphous compounds accompanying the sugar in sorghum. The Sugar Experiment Station of Louisiana has embodied in one of its bulletins the results of researches made in the destruction of glucose both in the sugarhouse and laboratory, also valuable chemical results obtained by Prof. Ross, of the State Experiment Station.

In the sugarhouses of Louisiana there were last year 53 chemists employed. Several of these made valuable contributions to the bibliography of sugar. A commission, consisting of the chief chemist of the Internal Revenue Bureau, the chief chemist of the Department of Agriculture, and the assistant in charge of weights and measures, appointed by Commissioner Mason to determine the proper standardization of polariscopes, and the methods for the optical estimation of sugars, and for securing uniform standards and methods, has recently made a most careful report, which it would be well for us to adopt intact as the official guide of this association. I therefore recommend that the methods now in use be modified as follows:

(1) Insert the words "sugar juices" before the word "molasses," in second line, under the methods of sugar analysis.

(2) Strike out all of section (*b*) in the method for the determination of water.

(3) Insert, as an alternate method for the estimation of ash, the Boyer method described on page 139 of Bulletin 28.

(4) On page 214 of Bulletin 28, line 7, add the words "or casserole" after the words "test tube."

(5) In the same section, after the words "acetic acid," insert "If preferred, the Ross method, using filter paper, may be used."

(6) Page 215, under *sucrose*, strike out section (*a*) entirely, and insert the report of the commission appointed by the Commissioner of Internal Revenue, for the purpose of establishing standards and methods for the polarimetric estimation of sugars.

(7) Strike out page 216 entirely, as it is given under methods for milk analysis, and is identically the same.

REPORT ON THE TRUE ASH IN PRODUCTS OF CANE SUGAR MANUFACTORIES, INCINERATED WITH SULPHURIC ACID.

By L. Biard.

The determination of ash in products of sugar manufactories is easily made according to the method of Scheibler, known as sulphuric incineration. It was proposed in 1865, in order to circumvent the inconveniences in the direct incineration proposed by Peligot in 1851, and carbonization with *titrage alkalimetrique* of the soluble salts proposed by Dubrunfaut in 1832. Scheibler having recognized that sulphuric incineration gave a greater weight of ash than by direct incineration, which might be calculated, proposed to apply to the sulphated ashes a coefficient for correction, which he fixed at 0.90, and which to-day is still generally employed. However, in 1870, Dubrunfaut drew attention to the fact that this coefficient of 0.90 could only be considered as a mean value, which might vary sensibly from the truth in many cases. He then proposed to add to the sugars finely powdered bakers' charcoal, or, better, platinum black. In the latter case complete incineration can be obtained in ten minutes at a low red heat. Dubrunfaut gives the following experiment: A sugar incinerated with platinum sponge gave 1.724 per cent of ash; by direct incineration 1.730; by sulphuric incineration, corrected by the coefficient 0.9, it gave 1.791. The first two results agree, giving a mean of 1.727, while in the third result the quantity obtained from a weight of sulphated ashes of $\frac{1.791}{0.9} = 1.99$. A true coefficient to convert the sulphated ash into true ash would thus be $\frac{1.727}{1.99} = 0.868$, and the coefficient 0.90 would be too high. This was the

conclusion arrived at by Vialette in a paper published in 1874, in which he concluded:

(1) The coefficient 0.9, adopted by the sugar industry for the analysis of sugars, is too high.

(2) In place of it, the coefficient 0.8 may be substituted with much greater exactness for crude sugars, excepting those of the first strike, for molasses of all sources, for diffusion liquids, and for the beet itself.

(3) For very pure sugars, such as those from the first strike, the value of the coefficient is still less, and should not be more than 0.7. These coefficients are not rigorously exact, and may vary within wide limits. For sugar beets, Viallette obtained 0.821 as a mean of ten experiments, with variations of from 0.75 to 0.87, and for normal molasses the mean of five experiments was 0.791, with variations of from 0.755 to 0.812. The cofficient 0.8, for sugar-beet molasses, is merely deduced from their normal ashes. According to Champion and Pellet (in 1874) they contain on an average, carbonic acid 28, chlorine 7, both of which are substituted by sulphuric acid. Now 28 of carbonic acid are equal to 50.91 of sulphuric acid, and 7 of chlorine equal to 7.89 of sulphuric acid, so that 28 + 7 equals 35, which, being replaced by 50.91 +7.89 =58.80, whence we obtain an increase of the weight of the ash by 23.80 per cent. One hundred parts of normal ash thus becomes 123.80; whence the proportion $\frac{100}{123.8}=0.808$, according to Champion and Pellet. But this reasoning is not entirely exact, for the sulphated ashes increase also by a quantity of oxygen equal to the chlorine, which, in the case given, would be 1.58. In fact the chlorides, in passing from the state of sulphates, not only have their chlorine replaced by sulphuric acid, but also take up one equivalent of oxygen to their metal. The true weight of sulphated ashes thus becomes $123.80 \times 1.58 = 125.38$, and the calculated coefficient is 7.97. So each of our observations is only of theoretical interest, and does not sensibly change the result of the calculation, owing to the small amount of chlorine. Be that as it may, the ordinary method of ash determination is defective, because it implies the application of an arbitrary coefficient, at least within certain limits, and it is also of interest to find a method permitting the rapid and convenient estimation of true ash.

Several years ago the use of vaseline was tried in Germany, but it does not appear to have given satisfactory results. More recently several processes have been proposed in France which appear to be more practicable. Jean Lucien incinerates in the presence of one-half per cent oxide of zinc (Bulletin de l'Association des Chimistes, February 15, 1889); J. de Robert uses oxalic acid in place of sulphuric acid, and destroys the last traces of carbon by nitrate of ammonia (Journal des Fabricantes de Sucre, July 3, 1889); finally E. Boyer employs benzoic acid in alcoholic solution (Bulletin de l'Association des Chimistes, February 15, 1890).

All these precautions are unnecessary when products of sugar-cane are to be incinerated, and we confine ourselves to burning at a low red heat, beyond which it is essential not to allow the temperature to rise in order not to volatilize chlorides. The substance, charred over a gas flame, is first placed on the edge of the muffle heated to a dull redness only on its farther portion. The ash becomes rapidly gray, and it is made almost white in putting it for a few minutes in the back portion of the muffle. We have noticed that the operation is more rapid in proceeding thus than by immediately putting the capsule into the maximum temperature, on account of the easier access of the air at the edge of the furnace. The incineration is more rapid than with sulphuric acid, requiring from thirty to forty-five minutes, and the ash presents a uniform slightly grayish tint without black points. The employment of a coefficient is thus rendered superfluous. We have, however, tried whether the coefficient 0.9 was too large also for sugar-cane products as it is for sugar-beet products. Our results gave the average of ten analyses for each product, and are given in a summary in the following table:

Proportion of true ash and sulphated ash in cane-sugar products.

Cane juice from first milling	0.812
Cane juice from second milling	0.822
Cane juice acidified by sulphurous acid	0.824
Cane juice defecated by sulphurous acid	0.777
Clarified juice from triple effect	0.807
Massecuite of cane juice (first strike)	0.787
Massecuite from first sirup (second strike)	0.767
Massecuite from second sirup (third strike)	0.770
Massecuite from third sirup (fourth strike)	0.767
Massecuite from fourth sirup (fifth strike)	0.779
Molasses	0.774
Sugar from second sirup (third strike)	0.777
Sugar from third sirup (fourth strike)	0.759
Sugar from fourth sirup (fifth strike)	0.781

The mean of all these coefficients, which vary but little from each other, is 0.785, which is sensibly the same as for sugar-beet products. We have tried if this result agrees with the composition of normal ash from these products. Without giving the complete analysis for each of these, which will be the subject of a future paper, we limit ourselves to giving the means of their composition:

Carbonic acid	6.36
Chlorine	12.64
Silica	2.87
Lime	16.76
Magnesia	9.38
Phosphoric acid	3.86
Sulphuric acid	17.73
Oxide of iron	2.36
Potash	30.88
Total	102.84
Oxygen corresponding to chlorine	2.84
Leaving	100.00

If the silica and iron are deducted, and we calculate on the one hand the acids as sulphuric acid, and on the other hand the quantity of sulphuric acid required to neutralize the bases, we find acids expressed as sulphurous acid 50.05, sulphuric acid required for neutralizing the bases 68.97; hence, when sulphuric incineration is practiced 68.87 — 50.05 = 18.92 of sulphuric acid are fixed in order to neutralize the alkalinity of the ashes. The increase in weight of the true ash may then be calculated as follows:

Loss of carbonic acid	6.36
Loss of chlorine	12.64
Loss of weight	19.00
Sulphuric acid equivalent to carbonic acid	11.56
Sulphuric acid equivalent to chlorine	14.24
Sulphuric acid required to neutralize the alkalinity	18.92
Oxygen equivalent to chlorine	2.84
Increase in weight	47.56
Loss in weight	19.00
True increase in weight	28.56

One hundred parts of true ash thus becomes 128.56 parts of sulphated ash, and the coefficient for calculating from the latter to the former is: $\frac{100}{128.56} = 0.778$. Hence this gives 0.7285, and it thus agrees with the calculation.

The recommendations of Mr. Stubbs were then taken up one by one and adopted.

Mr. STUBBS. I further recommended, on page 215, the number 144 be changed to 142.4.

Several members objected to making a change in the method of Clerget without any explanation of such change. It was finally agreed to make the change recommended, and in a footnote explain that 142.4 is the factor adopted by the association instead of 144, as given by Clerget.

Mr. STUBBS. At Mr. Crampton's suggestion I also recommend that under the method of Clerget, line 3, the word "strong" be struck out and the words "of 1.188 specific gravity", be inserted after the symbol "HCl." Carried.

Mr. HARPER. I wish to state that I have found a much better means of making determinations of invert sugars volumetrically than that usually practiced. By using a small flask instead of a casserole or test tube the heat is retained more constantly. If heated over an alcohol lamp in a 50 or 75 cc. Erlenmeyer flask the heat will be retained, and the work will be much more quickly and satisfactorily done.

Mr. STUBBS. I call attention to the fact that there is no method given for the determination of total solids or density, but it is something that has to be known in order to determine the purity coefficient of every juice and every sirup. If the association desires to adopt a method for this determination, I will prepare it for insertion in the forthcoming bulletin.

Mr. SHEPARD. I move that a method for this determination be inserted. Carried.

Mr. CRAWLEY. I ask for information as to the usual practice in determining ash from the juice of sorghum and sugar cane. The methods usually adopted are to evaporate the juice and then ignite it, or to evaporate and ignite with benzoic acid, or to ignite with sulphuric acid. It is well known that in ignition with sulphuric acid there is a great increase in weight, because the alkaline chlorides are changed into sulphates. On page 213, Bulletin 28, it is said under (*e*) "Saturate the sample with H_2SO_4, dry, ignite gently, then burn in muffle at low redness," but it does not say anything about subtracting a fixed per cent for this additional weight. In a great many determinations I have subtracted a tenth, and then determined the ash by the benzoic-acid method, but the results show, that if the benzoic-acid method be correct, one-tenth is much too small. I would like to know if others have had experience in this matter, and whether the method of subtracting one-tenth and determining with benzoic acid is usually pursued, or whether the sulphuric-acid method is usually used. I have found that the ash is more easily determined by the sulphuric-acid method.

Mr. STUBBS. I have a paper in French by Mr. Biard, which will be translated and embodied in my report, giving the results of a large number of analyses made by different chemists on this subject. We do not use sulphuric acid at all, because the exact amount to be deducted has not been determined.

The PRESIDENT. It would be well to strike it out, then.

Mr. STUBBS. Mr. Wiley has included it in the official method, but has given no deduction. After partially charring any sugar product and then adding the sulphuric acid, it would incinerate perfectly and give a beautiful white ash, and the rapidity of the operation is in its favor; and for refinery purposes, where absolute accuracy is not required, it is near enough, deducting one tenth, but in scientific work I do not use it at all. I ignite with benzoic acid, oxide of zinc, or alone. I find that saturating with hot water and then igniting gives very good results, though it is a little tedious.

Mr. CRAMPTON. This method of sulphated ash, as it is called, has a wide application in Europe, but I understand that such directions would probably not answer for cane, on which Mr. Stubbs had, of course, been using it. It would be well to make a distinction, if it is to be adopted, between its use on cane and on beet products.

The discussion of the report on sugar ended here, and the association proceeded to consider the next order of business.

REPORT ON PHOSPHORIC ACID.

By H. A. HUSTON.

The report, as heretofore, is divided into three sections:

Section 1 contains brief mention of abstracts of work published since the last meeting. This I have divided into two portions: (*a*) Work pertaining to the analytical processes; (*b*) work pertaining to the preparation of compounds of interest in connection with the study of phosphates. In the division relating to analytical work I have included a number of processes which were not originally intended for the determination of phosphates in fertilizers, but which seem from their nature to present points of interest to the fertilizer chemist.

I have also included the processes for the determination of iron and almina in phosphates, since this seems to be assuming a greater importance in connection with recent discoveries in this country.

The work relating to the determination of water in the superphosphates is also included under the division of analytical work.

The second section deals with the report of work done by chemists of the association cooperating with the reporter.

The third section deals with the recommendations for the ensuing year.

SECTION 1.—ABSTRACTS RELATING TO ANALYTICAL WORK.

Blum, L. Zeitschr. Anal. Chem., 29, 407. Abstract, Ber. Deut. Chem. Ges., 1890, 707 C.
Ueber den Nachweis fremder Rohphosphate in Thomas Schlackenmehl.

Breyer, Th., and Schweitzer, H. Oil Paint and Drug Reporter, March 25, 1891.
The determination of oxides of iron and aluminum in phosphates.

The authors have examined various methods for determining the oxide of iron and of aluminum in phosphates, including the conventional method so called, in which

the acid phosphate solution is rendered alkaline by ammonia and acidulated by acetic acid, or ammonia is added until the phosphate solution is nearly neutral, but still contains enough mineral acid to prevent the precipitation of phosphates.

Next an excess of acetate of ammonia or sodium is added, and the precipitate is filtered, washed, and weighed as phosphate of iron and aluminum. One-half of the weight obtained is stated as oxide of iron plus oxide of aluminum, or the total amount of phosphoric acid in the precipitate is determined and the oxides estimated by difference.

The authors criticise this method on account of the errors liable to arise from the presence of too much free acetic acid, or from the presence of fluorine, and also on account of the formation of basic phosphates in case the precipitate is washed with water.

The authors also refer to the method of Glaser, and to the work of Stutzer and Jones, preferring the modification of the Glaser method suggested by Jones, to the original Glaser method.

They also refer to the work of von Gruber in the same line. The authors, however, prefer to modify the Glaser method in the following manner: Take 100 cc. of the acid phosphate solution (prepared for the determination of phosphoric acid containing 5 gr. of substance in 500 cc.), transfer by means of a pipette into a 250 cc. flask, add 5 cc. sulphuric acid, specific gravity 1.84, allow it to cool, and add further 100 cc. of alcohol. Shake the flask occasionally during ten minutes, fill up to the mark and permit it to stand for half an hour; then fill up to the mark again, mix well, and filter. 200 cc. of the filtrate are then heated until the alcohol is evaporated, and the phosphates of iron and alumina are precipitated by rendering the solution alkaline by a slight excess of ammonia, this excess being removed by boiling. The precipitate is filtered off and washed with a hot neutral solution of nitrate or chloride of ammonium. This modification does not require the preparation of a special sample as proposed by Jones, and it increases the amount of substance from 0.4 gr. to 0.8 gr.; it also avoids the excess of sulphuric acid and increases the amount of alcohol, thus insuring a complete precipitation of the sulphate of lime. The application of a solution of nitrate or chloride of ammonium for washing the precipitate of the phosphates is to be demanded peremptorily.

The authors also call attention to the fact that many Carolina and Florida phosphates contain a certain amount of iron in combination with sulphur as pyrites; the amount of the iron is calculated in the results of the analysis as oxide of iron together with the iron that is contained in the phosphate as oxide. The authors state that pyrites is dissolved in the mixture of hydrochloric acid and nitric acid, or in hydrochloric acid and chlorate of potassium, but it is not acted upon by the hydrochloric acid or by the sulphuric acid as used in the manufacture of superphosphates. This iron, therefore, can not have any injurious effect upon the manufacture of superphosphate; for this reason the trade ought to insist that the amount of this iron be stated in the certificate of this analysis as oxide of iron from pyrites. The amount of this oxide of iron from pyrites may be ascertained by dissolving the phosphate in hydrochloric acid, and determining the iron contained in the insoluble residue.

Chappelle. Journ. de Pharm. (5), 21, 344. Abstract Journ. Chem. Soc., July, 1890, 825. Estimation of total phosphorus in urine.

Ten cc. are heated with 5 cc. of sulphuric acid until the liquid is decolorized. The solution is diluted to 50 cc., neutralized with sodium hydroxide, and precipitated with magnesia mixture.

Chatard, T. M. Amer. Chem. Journ., Vol. 13, 106. Journ. Anal. and Applied Chem., Vol. 5, 163. Abstracts Journ. Chem. Soc., 1891, 768. Ber. Deut. Chem. Ges., 1891, 331 C. Analyst, 1891, 129.

The separation of titanium, chromium, aluminum, iron, barium, and phosphoric acid in rock analysis.

In the process employed the details are of such importance that an abstract would be of little value.

Crispo, D. Zeitschr. Anal. Chem., 1891, 301. Abstract Chem. Centr.-Bltt., 1891, II, 220. Belgium method for the determination of water-soluble phosphoric acid in superphosphates.

The method is the same as used by the A. O. A. C., 1890-'91, except that the Belgian chemists use 5 grs. of superphosphate, and the washing is continued until the filtrate no longer gives a reaction for phosphoric acid; the volume is then made up to 500 cc., and the phosphoric acid determined by the molybdate method. In case the final washings cause a cloudiness, the precipitate is dissolved by adding a little nitric acid before making up to 500 cc. The author gives reasons why this method is preferred to the "digestion method," formerly used by the Belgian chemists. The new method has been in use for two years in Belgium.

Dancy, A. B. Proc. A. O. A. C., 1890, Chem. Div. U. S. Dept. Agr. Bul. No. 28. Journ. Anal. and Applied Chem., Vol. 4, 395.

The determination of available phosphoric acid in fertilizers containing cotton-seed meal.

Fresenius, W. Zeitschr. Anal. Chem., 28, 87. Abstract. Journ. Anal. and Applied Chem., Vol. 4, 86.

Contribution to the determination of phosphoric acid in sweet wines and notes on their critical examination.

Gibson, C. A. Proc. A. O. A. C., 1890, 166.

Differences in the determination of reverted phosphoric acid by the official method of analysis.

Glaser, E. Zeitschr. Angew. Chem., 89, 636. Abstract Journ. Anal. and Applied Chem., Vol. 4, 69.

Estimation of iron and alumina in phosphates.

For later modification of this method, see R. Jones, and also H. H. B. Shepherd and Breyer.

Goessman, C. A. Report Mass. State Agr. Expt. Station, 1890, 272. Oil, Paint, and Drug Reporter.

Methods of fertilizer analysis.

Preparation of sample.—The entire available sample is spread upon a smooth, hard surface, and intimately mixed without grinding, all lumps being broken up with a spatula. Unnecessary loss or gain of moisture is to be avoided. Moisture: Dry 2 grs. in the air-bath at 100° C. to 110° C. to a constant weight.

Total phosphoric acid.—Weigh out 2 grs. in a platinum crucible, and destroy the organic matter by carefully burning in a muffle. Weigh when cool, to determine the "organic and volatile matter." Digest the crucible and contents with dilute hydrochloric acid until solution is complete. Filter and evaporate the filtrate to complete dryness. The "insoluble matter" on the filter is burned and weighed. The residue left from the evaporation is taken up with dilute nitric acid, if the molybdic method is to be followed, but with hydrochloric acid if method (2) is preferred. The solution after filtering is made up to a volume of 200 cc. with distilled water.

(1) The molybdic method: 25 cc. of the solution are digested in a water bath at 65° C. from one to two hours, with an excess of molybdic solution. The precipitate is brought upon a filter and washed with water containing a little molybdic solution. It is then dissolved in ammonia water, the solution nearly neutralized with hydrochloric acid, and magnesia mixture added slowly, with constant stirring. The precipitate is allowed to stand at least three hours, when it is filtered through a Gooch crucible, washed with dilute ammonia, ignited, and weighed.

(2) The following method is occasionally employed when phosphates of iron and alumina are present in small quantities only: To 50 cc. of the hydrochloric acid solution add ammonia in slight excess. After standing a few minutes, acidify with acetic acid and filter off the phosphates of iron and alumina, washing carefully with water. To the filtrate add sufficient oxalate of ammonia to precipitate all the lime; digest

for several hours at a temperature below boiling, and filter through double filters which have previously been washed with oxalate of ammonia, washing thoroughly with water. Dissolve the phosphates of iron and alumina on the filter with warm dilute hydrochloric acid, and wash into a beaker containing a small quantity of powdered tartaric acid. When the latter has gone into solution, mix with the filtrate from the oxalate of ammonia. The phosphoric acid is precipitated with magnesia mixture, and treated as in (1).

Soluble phosphoric acid.—Weigh out 2 gr. into a beaker, cover with 10 to 15 cc. of water, and allow it to stand for fifteen minutes, stirring three times at equal intervals. Decant the solution through a filter into a graduated cylinder. Add another like quantity of water and let it stand fifteen minutes more, stirring as before. Filter the solution into the cylinder, and wash the residue on the filter until the filtrate amounts to 200 cc. The phosphoric acid is determined in an aliquot part of the solution as under total phosphoric acid.

Insoluble phosphoric acid.—Add 100 cc. of neutral ammonia citrate (sp. gr. 1.09) to the beaker in which the digestion with water has been made. Put in a water-bath and heat to 65° C. Drop in the filter containing the residue from the above operation, and digest for thirty minutes, stirring every five minutes. Filter and wash thoroughly, using the suction pump. Dry and burn. The ash is then treated as under total phosphoric acid.

Reverted phosphoric acid.—The sum of the soluble and insoluble subtracted from the total gives the reverted or citrate-soluble phosphoric acid.

Reagents.—The reagents used in the estimation of phosphoric acid are prepared according to directions given in the Proceedings of the Association of Official Agricultural Chemists, 1889, pp. 225 and 226.

Gruber, v. Zeitschr. Anal. Chem., 30, 9. Abstracts Journ. Chem. Soc., 1891, 561. Ber. Deut. Chem. Ges., 1891, 475 C.

Estimation of ferric oxide and alumina in phosphates.

Gruber, v. Zeitschr. Anal. Chem., 30, 206. Abstracts Journ. Chem. Soc., 1891, 963. Ber. Deut. Chem. Ges., 1891, 583 C.

Jones's modification of Glaser's method for the estimation of ferric oxide and alumina in phosphates.

Holverscheit, R. Chem. Centr.-Bltt., 1890, I, 977. Abstracts Journ. Chem. Soc., 1890, 1343.

Quantitative estimation of vanadium, and separation of vanadic acid from phosphoric acid.

Hundeshagen, F. Zeitschr. Anal. Chem., 28, 141. Chem. News, Vol. 60, 168. Abstract Journ. Anal. and Applied Chem., Vol. 4, 60.

Phospho-dodeka-molybdic acid.

A study of the conditions of the formation of yellow phospho-molybdic acid, and its separation as ammonium phospho-molybdate.

Huston, H. A. Proc. A. O. A. C., 1890, 170.

Action of ammonic citrate on high grade aluminic phosphate.

Johnson, S. W., and Osborn, T. B. Report Conn. Agr. Expt. Station, 1890, 195.

On the determination of phosphoric acid in presence of iron and alumina.

The authors show that in the presence of iron and alumina the present official method gives too high results. They also show that the original molybdate method as worked out by Sonnenschein (solution in sulphuric or nitric acid, addition of excess of molybdate solution to the cold solution of the phosphate, standing four to six hours near but not exceeding 50° C., washing with dilute molybdate solution) gives correct results. They state that the error is caused by iron and alumina contaminating the yellow precipitate, and afterward passing into the ammonium magnesium phosphate. They also state that in presence of excess of nitric acid or relative deficiency of the molybdate solution, all the phosphoric acid may not enter the precipitate. Results are given by three workers on 8 samples, using two sets of re-agents.

Jones, C. Chem. News, 62, 220. Journ. Anal. and Applied Chem., Vol. 4, 268. Trans. A. I. M. E. Abstract Journ. Chem. Soc., 1891, 363.

Phosphorus in pig-iron, steel, and iron ore.

The author quotes authorities, and advocates oxidation with permanganate, washing the molybdate precipitate with sulphuric acid and ammonium sulphate, the use of ferrous sulphate instead of an organic acid to redissolve the manganic hydroxide. The article is of interest in connection with the method proposed by Dr. De Roode for determining both phosphoric acid and nitrogen in the same sample.

Jones, R. Chem. Zeit., 14, 269. Abstracts Journ. Anal. and Applied Chem., Vol. 4, 214, Analyst, 1891, 39. Chem. Centr.-Bltt., 1891, 1, 280.

Determination of oxides of iron and aluminum in phosphates.

Estimation of iron and alumina in phosphates. R. Jones (Zeitschr. Angew. Chemie, No. 1, 1891).—The author having thoroughly tried Glaser's process (see Analyst, 1890), thinks it may be improved by lessening the amount of sulphuric acid. The original process recommends 4 gr.–5 gr. of the strongest acid, but even if the sample solely consisted of calcic oxide, 2 gr. would be amply sufficient. As the measuring of strong sulphuric acid is troublesome, the author uses 10 cc. of an acid which has been five times diluted. The author further thinks the quantity of phosphate used for analysis (0.4 gr.) is too small; at least 1 gr. should be used. The actual process is then as follows:

"Ten gr. of the phosphate are dissolved in nitro-hydrochloric acid, and the solution diluted up to 500 cc. 50 cc. (1 gr.) are evaporated to half the bulk, and while still hot mixed with 10 cc. of dilute sulphuric acid. 150 cc. alcohol are next added, and the mixture allowed to settle for at least three hours. Glaser thought half an hour sufficient, but the author found this to be not long enough. The sulphate of lime is collected on a filter and washed with alcohol, the filtrate being collected in an Erlenmeyer's flask of half a liter capacity. The washing is finished when 10 drops of the washings, after dilution with an equal bulk of water, do not color with methyl-orange. The filter and the precipitate are put into a platinum dish, the spirit is burned off, and the mass finally ignited and weighed.

"The alcoholic solution is distilled off to recover the alcohol (which, however, must be redistilled over potash), and the residue rinsed into a beaker. Slight excess of ammonia is then added, and this again completely boiled off. This is very important, as it prevents the co-precipitation of magnesia.

"The precipitate, consisting of ferric and aluminic phosphates, is carefully collected on a filter and washed four times with boiling water without disturbing it too much. If the washings should be turbid, washing with a very weak solution of neutral ammonium nitrate must be resorted to. The filtrate contains phosphoric acid and magnesia besides, of course, sulphuric acid, and, maybe, alkalies. The magnesic phosphate is separated by addition of ammonia and weighed, but the phosphoric acid and alkalis are best estimated in aliquot parts of the original solution.

"Many analysts prefer to remove the phosphoric acid by molybdate, and finally weigh the pure oxides. The author thinks this a very good plan, providing the phosphates of iron and alumina have been first isolated by Glaser's process before treating with molybdate. The phosphate must, however, be free from organic matter, otherwise a little of the alumina will escape precipitation."

Martinotti, Federico: Le Stazione Speriment. Agric. Ital. Abstract Chem. Centr.-Bltt., 1891, 1, 732.

The determination of phosphoric acid in manures.

A citrate method, preceded by the removal of nitric acid by repeated evaporations with hydrochloric acid.

Meyer, D. Th. Chem. Zeit., No. 102, 1890. Abstracts Analyst, 1891, 60. Ber. Deut. Chem. Ges., 1891, 47 C.

Note on Glaser's process for the estimation of iron and alumina in manures.

A joint committee of German manure-makers and agricultural analysts, assembled last year in Bremen, has reported in favor of Glaser's spirit method, instead of the ammonia and acetic acid process. That process can not very well be defended. If too much acetic acid is added the results are too low, and if the manure contains a fluoride they will be too high. These sources of error do not occur in Glaser's process. The separation of the iron and alumina from the lime is very successful, and allows the latter to be very accurately determined. The gypsum takes, however, some time for complete separation. The drawback to Glaser's process is his complete ignoring of the magnesia. True, its sulphate is practically insoluble in absolute alcohol, but far from insoluble in a mixture of alcohol, water, and sulphuric acid. A fluid prepared by shaking 25 cc. of absolute alcohol, 20 cc. of water, and 5 cc. of sulphuric acid, with powdered Epsom salts, contained 3.2 grams of magnesia per 250 cc. Suppose the sample to consist of pure magnesia, every trace of it would remain in the alcoholic fluid. Another experiment convinced the author of the co-precipitation of the magnesia with the iron and alumina. He twice analyzed a solution of lime, iron, and aluminic phosphates, but in one case added sulphate of magnesia. The separated sulphate of lime proved to be pure, but in the second experiment the precipitated iron and alumina phosphates weighed three times heavier than in the first one. And a qualitative analysis proved this to be due to the presence of magnesia.

It is therefore not a matter of astonishment that Glaser's method gives, as a rule higher results than the conventional method, even if the manure should contain fluorides.

To see in how far the process can be trusted for commercial purposes, the author tried several Belgian phosphates. The magnesia was each time estimated by redissolving the precipitate in hydrochloric acid, boiling with some ferric chloride, and precipitating the excess of this, together with phosphoric acid, with ammonia. The magnesia was then precipitated as usual with phosphate of soda.

Sample No. 1.—Obtained 0.63 per cent of mixed iron and alumina; but, after estimating the magnesia, this became reduced to 0.48 per cent.

Sample No. 2.—Obtained 4.23 per cent. After allowing for magnesia, only 4.15 per cent.

Sample No. 3.—Obtained 3.52 per cent. After allowing for magnesia, 3.12 per cent.

Of course, if the amount of magnesia in the manure is very trifling, no harm will be done; but if large, it must seriously interfere with the accuracy of the process. Glaser's method is therefore not yet fit to become the recognized trade process, and is, in fact, at the moment not much better than the old conventional one, unless one takes the trouble to estimate the co-precipitated magnesia.

Oliveri, V. Le Stazione Speriment. Agr. Ital., febbrajo 1891. Abstract Journ. Anal. and Applied Chem., Vol. 5, 415.

Estimation of phosphoric acid in basic slags.

A weighed quantity of the slag is reduced to a fine powder. To 5 gr. of the sample are added three times its weight of chlorate of potash, and the whole is intimately mixed. The mixture is then placed in a porcelain dish, and hydrochloric acid is added, little by little, until the potash is completely decomposed. Evaporation is then carried on until the mass is dry. The material is then treated with fuming nitric acid, and the determination of the phosphorus is made by the ordinary gravimetric method. This is accomplished by taking the liquid portion of the solution and precipitating the phosphoric acid as ammonium magnesium phosphate. After washing, it is finally weighed as pyrophosphate of magnesia.

By carrying on the operation as described above a reduction of phosphoric acid is avoided, and the presence of an abundant quantity of potash prevents the formation of basic phosphate of iron, which is insoluble in nitric acid.

Reinhardt, C. Chem. Zeit., 1891, xv, 410. Abstract, Ber. Deut. Chem. Ges., 1891, 407 C. Chem. Centr.-Bltt., 1891, i., 843.

Zur Bestimmung des Phosphors im Roheisen mittelst der Braun'schen Eimercentrifuge.

Shepherd, H. H. B. Chem. News, 63, 251. Abstract Journ. Anal. and Applied Chem., Vol. 5, 418. Chem. Centr.-Bltt., 1891, II, 86.

The Glaser or alcohol method for determination of iron and alumina in phosphates (modified).

The author gives details of the method, introducing modifications which meet objections brought against this method in England. 2.5 grs. of the phosphate are dissolved in 10 cc. hydrochloric acid, evaporated to dryness, taken up with hydrochloric, raised to boiling, washed out into a 250 cc. flask with as little water as possible. 10 cc. concentrated sulphuric acid are now added, and the solution is allowed to stand for five minutes, with frequent shaking. 95 per cent alcohol is now added, the mixture is cooled, made up to the mark with alcohol, and well shaken. After the contraction in volume which takes place, it is again made up to 250 cc. and mixed. After standing one hour it is filtered and 200 cc. (2 grs. phosphate) are taken and evaporated to a small bulk. When organic matter is present, it is desirable to evaporate to pastiness, that the acid may partially decompose it. The solution is now washed into a beaker with about 50 to 100 cc. of water, boiled for a short time with bromine or other oxidizing agent, and after adding ammonia, it is again boiled for about half an hour and cooled; a little more ammonia is added, filtered, washed with a hot solution of ammonium chloride, ignited and weighed. The ammonium chloride is used to prevent the precipitate from washing through the filter. The phosphoric acid is determined by dissolving the ignited precipitate in acid, and the oxide of iron and alumina obtained by difference. Messrs. Teschemacher and Smith add the postscript, that when magnesia is present, the phosphates of iron and alumina obtained as above must be freed from this impurity by washing the precipitate off the filter, and boiling with water and a little nitrate of ammonium.

Stocklasa, J. Zeitschr. Anal. Chem., 1890, 390. Chem. News, Vol. 63, 101. Abstracts Journ. Anal. and Applied Chem., Vol. 5, 475. Ber. Deut. Chem. Ges., 1890, 706 C.

Determination of water in superphosphates.

Experiments were made upon a chemically pure monocalcium phosphate at temperatures of 100° C., 105–120° C., 150°, and 200° C., and the decompositions produced at temperatures above 105° C. were examined into.

Stutzer, A. Zeitschr. Angew. Chem., 1890, 43. Abstracts Journ. Chem. Soc., 1891, 245. Journ. Anal. and Applied Chem., Vol. 4, 215.

Estimation of ferric oxide and alumina in phosphatic manures.

The method consists in precipitating the iron, alumina, and phosphoric acid in an ammonium acetate solution, filtering and treating the precipitate with molybdate solution, when the phosphoric acid is precipitated. This is filtered, and the iron and alumina precipitated from the filtrate by ammonia.

Vogel, J. H. Zeitschr. Angew. Chem., 1891, 357. Abstract Chem. Centr.-Bltt., 1891, II, 224.

Die Bestimmung des Kalkes in Phosphaten bei Anwendung der Glaser'schen Methode.

Wiley, H. W., sec'y. Proc. A. O. A. C., 1890, 228.

Official method for determining phosphoric acid.

Williams, W. J. Proc. A. O. A. C., 1890, 177.

Citrate soluble phosphoric acid in aluminic phosphate. Compilation of work by various chemists.

ABSTRACTS RELATING TO GENERAL WORK WITH PHOSPHATES.

Causse, H. Journ. de Pharm. (5) 21 544–547. Abstract Journ. Chem. Soc., 1890, 1056.

Preparation and stability of dicalcium phosphate.

Causse, H. Bull. Soc. Chim. de Paris, March 1891, 298.

Sur le dédoublement du phosphate mono-calcique par les acétates.

Haack, K. Chem. Centr. Bltt., 1890, II, 736–737. Liebig's Ann., 262, 189. Journ Chem. Soc., 1891, 400.

Mercury arsenates and phosphates.

Maercker, M. Magd. Zeit., 1890. Abstract Bied. Centr.-Bltt., 20, 4–11. Journ. Chem. Soc., 1891, 612. Chem. Centr. Bltt., 1891, I, 1065.

Economy of phosphoric acid in the growth of the beet root.

Merz, J. Chem. Zeit., 14, 95. Abstract Journ. Chem. Soc., 1891, 106.

Amount of fat in bone meal.

The author considers the extraction experiments of Stocklasa defective and quotes extraction experiments of his own in which the fat was almost completely removed.

Nicolas, M. Compt. Rend., 111, 974–975. Abstract Journ. Chem. Soc., 1891, 398. Ber. Deut. Chem. Ges., 1891, 68 C. Chem. Centr. Bltt., 1891, I, 253.

Preparation of pure phosphoric acid.

Pointet, G. Bull. Soc. Chim. de Paris, Feb., 1891, 255.

De la composition du biphosphate de chaux commercial dans ses diverses formes, et de la préparation du biphosphate de chaux cristallisé.

Stocklasa, J. Landw. Vers.-Stat., 38, 401–410. Abstract Journ. Chem. Soc., 1891, 880. Ber. Deut. Chem. Ges., 1890, 626 C.

The soluble phosphoric acid compounds of superphosphates.

Stocklasa, J. Chem. Zeit., 14, 1–2; 21, 32–33. Abstract Journ. Chem. Soc., 1891, 105.

Composition of bone meal.

A paper showing the effects of digesting ground bone with water, and steaming under pressures between 0.5 and 2.5 atmospheres; also results of extracting with petroleum under various pressures, and expelling petroleum with steam.

The author considers that the apparent failure of bone meal as a manure in certain cases is due to neglect of certain points set forth in his paper.

Stocklasa, J. Chem. Zeit., 13, 203–210, 240–243, 273–283. Abstract Journ. Chem. Soc., 1890, 695.

Preparation and solubility of monocalcium phosphate.

Thompson, A. Bied. Centr. Bltt., 19, 585–588. Abstract Journ. Chem. Soc., 1891, 105.

Behavior of sandy soils toward superphosphates.

A paper relating to laboratory experiments on soil absorption for superphosphates.

Winsinger, C. Abstract, Ber. Deut. Chem. Ges., 1890, 54 C.

Method for the preparation of anhydrous phosphate precipitate ble in ammoniacal ammonium citrate. (Patent specifications.)

Section 2.—Report of the work done on samples sent out by the reporter.

On February 11 letters were sent to sixty-fourche mists, asking them if they would be willing to take part in the work upon phosphoric acid, and stating that the sentiment at the last meeting of the association was that only those should be asked to take part who had special interest in, or knowledge of, the subject. Forty-one replies were received, and thirty-four chemists consented to take part in the work if possible. Twenty-five chemists sent reports, the results of which appear in the table below. The samples were: No. 1, a mixture in about equal proportions of dissolved bone black and cotton-seed meal; No. 2, ground bone; No. 3, high grade aluminic phosphate.

Results by official methods only.

Analyst.	No. 1.						No. 2.				No. 3.				Remarks.
	Water.	Phosphoric acid.					Water	Phosphoric acid.			Water	Phosphoric acid.			
		Soluble.	Reverted.	Available.	Insoluble.	Total.		Citrate soluble.	Insoluble.	Total.		Citrate soluble.	Insoluble.	Total.	
	P. ct.	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	
E. H. Farrington, Champaign, Ill	10.00	8.00	2.15	10.15	1.35	11.50	5.44	11.71	15.04	26.75	5.65	33.55	11.25	44.80	
George L. Teller, Fayetteville, Ark	10.34	7.91	1.63	9.54	1.57	11.11	5.43	7.84	18.07	25.91	5.69	28.94	15.83	44.77	
R. B. Moore, Amherst, Mass	12.81	8.31	2.38	10.69	0.77	11.46	5.84	7.36	18.81	26.17	6.86	23.08	20.88	43.96	Water determined in air bath at 100° C.
F. W. Morse, Hanover, N. H	10.10	7.90	2.61	10.51	1.34	11.85	5.53	10.39	16.06	26.45	6.25	28.39	14.88	43.27	Water determined in air bath at 100° C.
C. B. Collingswood, Tucson, Ariz	10.90	7.51	2.71	10.22	1.16	11.38	.....	.....	.....	.....	.....	.....	.....	.....	Water determined in bath with brine at 100° C.
R. De Roode, Morgantown, W. Va	9.70	7.70	2.03	9.73	1.38	11.28	5.77	3.79	22.30	26.09	6.21	27.48	16.81	44.29	Water determined by heating five hours at 101° C. Citrate neutralized by J. A Myers.
B. H. Hill, Morgantown, W. Va	9.60	7.84	2.01	9.85	1.40	11.25	5.72	2.97	22.95	25.92	6.41	28.70	15.00	43.70	Water determined by heating five hours at 101° C. Reagents by J. A. Myers.
R. C. Kedzie, Agricultural College, Mich.	11.27	7.16	2.81	9.97	1.31	11.28	5.28	8.20	18.30	26.50	5.90	27.82	15.85	43.67	
Harry Snyder, Ithaca, N. Y	.....	.....	.....	.....	.....	.....	5.91	.....	.....	26.34	6.32	21.16	23.37	44.53	Asbestos filter for citrate soluble.
A. E. Knorr, U. S. Department of Agriculture.	9.01	8.12	1.84	9.94	1.50	11.46	5.17	14.25	11.76	26.01	5.45	25.37	18.38	43.75	
A. B. McDonnell and J. A. Fries, State College, Pa.	10.01	7.75	2.49	10.24	1.27	11.51	5.43	8.66	17.96	26.62	5.86	30.61	14.50	45.11	

Results by official methods only—Continued.

Analyst.	No. 1.						No 2.				No. 3.				Remarks.
	Water.	Phosphoric acid.					Water	Phosphoric acid.			Water	Phosphoric acid.			
		Soluble.	Reverted.	Available.	Insoluble.	Total.		Citrate soluble.	Insoluble.	Total.		Citrate soluble.	Insoluble.	Total.	
	P. ct.	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	
N. W. Lord, Columbus, Ohio	9 17	8. 06	2. 02	10. 08	1. 21	11. 29	5. 45	9. 46	16. 70	26. 16	5. 90	30. 79	13. 56	44. 35	
J. S. Meng, Agricultural College, Miss	.....	8. 05	1. 73	9. 78	1. 48	11. 26	.....	8. 79	17. 35	26. 14	.....	22. 82	20. 73	43. 55	
F. S. Shiver, Fort Hill, S. C	.....	.....	.....	.....	.....	11. 08	.....	.....	.....	26. 38	.....	.....	.....	44. 14	
J. M. Bartlett, Orono, Me	.....	7. 65	2. 40	10. 05	1. 30	11. 35	.....	7. 38	18. 88	26. 26	.....	29. 16	14. 47	43. 63	
L. H. Merrill, Orono, Me	.....	7. 93	2. 28	10. 21	1. 16	11. 37	.....	6. 63	19. 90	26. 53	.....	.....	.....	44. 40	
H. E. Curtis, Lexington, Ky	9. 38	7. 63	2. 19	9. 82	1. 40	11. 22	5. 31	.....	.....	26. 24	5. 75	24. 97	19. 14	44. 11	
J. W. Wilkinson, Auburn, Ala	11. 37	7. 46	2. 59	10. 05	0. 97	11. 02	5. 07	13. 65	12. 75	26. 40	.....	.....	.....	.....	
W. J. Gascoyne, Baltimore, Md	9. 75	8. 01	2. 04	10. 05	1. 20	11. 25	5. 40	9. 84	16. 67	26. 51	5. 90	30. 29	14. 61	44. 90	
H. B. McDonnell, College Park Md	10. 15	7. 83	2. 25	10. 08	1. 31	11. 39	5. 40	7. 78	17. 96	25. 74	5. 69	29. 64	14. 50	44. 14	
Geneva Experiment Station, N. Y., W. H. Tibballs, P. H. Seymour.	9. 38	8. 06	1. 93	9. 99	1. 48	11. 47	5. 34	12. 55	14. 25	26. 80	5. 92	23. 33	21. 83	45. 16	
B. W. Kilgore, Raleigh, N. C	.....	7. 70	2. 26	9. 96	1. 27	11. 23	.....	9. 39	16. 86	26. 25	.....	23. 75	19. 78	43. 53	
—— Carpenter, Raleigh, N. C	.....	7. 67	2. 18	9. 85	1. 32	11. 17	.....	9. 05	17. 17	26. 22	.....	27. 85	15. 91	43. 76	
H. A. Huston, La Fayette, Ind	9. 75	7. 82	2. 24	10. 06	1. 26	11. 32	4. 63	8. 78	17. 57	26. 35	4. 66	25. 51	19. 62	45. 13	Total of No. 3 tested for Al in ignited pyrophosphate. None found.
Walker Bowman, 76 Broad street, New York City.	10. 12	8. 45	2. 26	10. 71	1. 17	11. 88	5. 22	8. 63	19. 11	27. 74	5. 19	34. 09	11. 31	45. 40	
Means	9. 82	7. 85	2. 22	10. 06	1. 29	11. 35	5. 41	8. 91	17. 45	26. 35	5. 86	27. 49	16. 77	44. 26	
Difference between highest and lowest.	3. 80	1. 29	1. 18	1. 17	0. 80	0. 86	1. 28	11. 28	11. 19	2. 00	2. 20	12. 93	12. 12	2. 13	
This difference expressed in per cent of average.	38. 60	16. 40	53. 10	11. 60	62. 00	7. 60	23. 70	126. 60	64. 10	7. 60	37. 50	47. 00	72. 30	4. 50	

Difference between highest and average.	2.99	0.60	0.59	0.65	0.28	0.53	0.50	5.34	5.50	1.39	1.00	6.60	6.60	1.14
This difference expressed in per cent of average.	30.60	7.60	26.60	6.40	21.70	4.70	9.20	59.90	31.50	5.20	17.10	24.00	38.90	2.50
Difference between lowest and average.	0.81	0.69	0.59	0.48	0.52	0.33	0.78	5.94	5.69	0.61	1.20	6.33	5.52	0.99
This difference expressed in per cent of average.	8.20	8.80	26.60	4.80	40.30	2.90	14.40	66.60	32.60	2.30	20.50	23.00	33.40	2.00

In tabulating these results and taking averages, nothing has been excluded. The summaries at the foot of the table are treated according to the method which has been used by the reporter for cattle food in previous years. It will be noticed that in this work results which are fairly satisfactory from a trade standpoint appear very bad according to the standard set by the association, in which the error should not exceed 10 per cent of the total quantity involved. Thus, in the table under "insoluble," for No. 1, I think that the results would all be considered fairly good, while the difference between the highest and the lowest comes to 61 per cent of the total amount involved, the departure from the mean to the lowest being 40 per cent, and from the mean to the highest 25 per cent of the amount involved. On the other hand, on the column marked total under No. 3, the percentage of difference between the highest and lowest is well within the standard set by the association, while the real error 2.13 per cent is altogether too large to be overlooked.

The samples were prepared according to the official method, that is, all the material sent out was passed through a sieve with round holes 1 mm. in diameter. I think that heretofore many of the samples sent out have been passed through sieves which were smaller than those recommended by the association, and your reporter deemed it best to use the standard sieve for this work. The samples were thoroughly mixed after sifting.

WATER.

The results of the different analyses upon the water contained in these samples is far from satisfactory. In many cases the official method was not adhered to, and even where it was, the results have varied quite widely. I think it quite remarkable that such varying results should have been obtained upon a material like No. 3, or even upon a material like ground bone, which ought to have been fairly uniform in its water contents. The samples were all put up at the same time, and after having been exposed long enough to the air of the room, should have been in a fairly stable condition as regards the moisture present. A few workers made some special investigations in regard to the amount of water, as determined by the different methods. The results of these determinations appear in the following table:

Special table of determinations of water.

Investigator.	Method.	No. 1.	No. 2.	No. 3.
		Per ct.	*Per ct.*	*Per ct.*
Farrington	Official	10.00	5.44	5.65
	2 gr. heated in dry hydrogen	12.75	6.60	7.65
De Roode	5 hours at 101° C	9.70	5.77	6.21
	10 hours at 101° C	10.39	5.79	6.33
Knorr	Official	9.01	5.17	5.45
	4 hours in hydrogen	10.29	6.49	6.73
	12 hours in hydrogen	11.12	Gain.[1]	Gain.[1]
	5 hours at 105° C	9.86	5.35	5.82
Van Slyke	Moisture increased on standing:			
	First determination	9.38	5.34	5.92
	Second determination	9.59	5.63	6.24
	Third determination	9.87	5.68	6.29
Huston	5 hours in steam oven at 100° C	9.75	4.63	4.68
	9 hours in steam oven at 100° C	10.13	5.01	4.85
	13 hours in steam oven at 100° C	10.48	5.17	5.28
	17 hours in steam oven at 100° C	10.61	Gain.	5.38
	21 hours in steam oven at 100° C	10.71	Gain.	Gain.
	29 hours in steam oven at 100° C	11.15	Gain.	Gain.
	5 hours in hydrogen in steam bath	11.01	5.98	6.69
	9 hours in hydrogen in steam bath	11.98	6.24	Gain.
	13 hours in hydrogen in steam bath	12.11	Gain.	Gain.

[1] Gain after 8 hours.

I think that the most remarkable feature of this table is the results obtained by Mr. Knorr and myself, from which it appears that both No. 2 and No. 3 showed a decided gain in weight after drying some hours in hydrogen. In most cases it will be noted that the dryings were continued for a long time after the first gain was apparent, and still the gain did not disappear. I believe that this gain is not due to any impurities or lack of drying in the hydrogen, because the hydrogen itself in both cases was submitted to such thorough drying and preliminary purification, that I do not see how any moisture could have been carried forward. The apparatus used by Mr. Knorr and myself for this work was practically the same, and was that figured in last year's work, Bull. 28, Chem. Div. U. S. Dept. of Agr., p. 100. There is no question but that the determination of water in fertilizers should receive much fuller and more careful investigation, and your reporter regrets that the time at his disposal did not permit of a more extended investigation of this matter.

SOLUBLE PHOSPHORIC ACID.

I think the results upon soluble phosphoric acid are to be considered fairly satisfactory. If we go further than this, and take the amount of available phosphoric acid in No. 1, the results appear even more satisfactory, and I think that upon this class of goods the methods ought to be considered as reasonably exact.

TOTAL PHOSPHORIC ACID.

The determination of total phosphoric acid in No. 1 and No. 2 are, with a single exception, fairly satisfactory, and even in the exceptional cases the differences are not great. In No. 3 the results are as good as could be expected from the official methods which were in use last year. With the additional methods adopted this year, I believe that a decided improvement in the results will follow for this class of goods.

Many of the chemists cooperating with the reporter did special work upon the total phosphoric acid, the results of which appear in the following table:

Analyst.	Sample No.	Nitric and hydrochloric acid.	Hydrochloric acid and potassium chlorate.	Mg. nitrate, solution in nitric acid.	Mg. nitrate, solution in hydrochloric acid.	Mg. nitrate, solution in nitric and hydrochloric acid.	Nitric and hydrochloric acid, prec. at 40° C., stood at air temperature.	Aqua regia, prec. at 40° C., stood at air temperature.	Sulph. acid, Hg or HgO, and permanganate (De Roode).	Sulph. acid, H_2O, permanganate and ferrous am. sulph. before precipitating.	Mg. nitrate, solution in nitric acid, silica removed.	Sulph. acid, and potassium or sodium nitrate (Burney).
		P. ct.	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*
E. H. Farrington	1	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....
	2	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....
	3	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....
George L. Teller	1	.....	.....	11.11	.....	.....	.....	.....	.....	.....	.....	.....
	2	25.91	26.03	.....	.....	.....	.....	.....	.....	.....	.....	.....
	3	43.22	44.77	44.51	.....	.....	.....	.....	.....	.....	.....	.....
R. B. Moore	1	.....	.....	.....	11.46	.....	.....	.....	.....	.....	.....	.....
	2	.....	.....	.....	26.17	.....	.....	.....	.....	.....	.....	.....
	3	43.96	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....
F. W. Morse	1	.....	.....	.....	.....	11.85	.....	.....	.....	.....	.....	.....
	2	26.45	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....
	3	43.27	.....	.....	.....	.....	44.03	44.92	.....	.....	.....	.....
C. B. Collingwood	1	.....	.....	[1]11.38	[1]11.38	.....	.....	.....	.....	.....	.....	.....
R. De Roode	1	.....	.....	.....	.....	.....	.....	....	11.11	.....	.....	.....
	2	.....	26.09	.....	.....	.....	.....	.....	26.59	.....	.....	.....
	3	44.29	.....	.....	.....	.....	.....	.....	44.31	.....	.....	.....
B. H. Hill	1	.....	.....	[1]11.25	[1]11.25	.....	.....	.....	11.37	.....	.....	.....
	2	.....	.....	25.92	25.92	.....	.....	.....	26.02	.....	.....	.....
	3	43.95	.....	.....	.....	.....	.....	.....	44.67	.....	.....	.....
R. C. Kedzie	1	11.04	10.95	11.28	.....	.....	.....	.....	.....	.....	11.24	.....
	2	25.35	26.50	.....	.....	.....	.....	.....	.....	.....	.....	.....
	3	43.67	43.61	.....	.....	.....	.....	.....	.....	.....	.....	.....
H. Snyder	2	26.34	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....
	3	44 53	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....
A. E. Knorr	1	.....	.....	.....	.....	.....	.....	.....	.....	.....	11.22	.....
	2	.....	.....	.....	.....	.....	.....	.....	.....	.....	25.66	.....
	3	43.75	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....
J. A. Fries	1	.....	.....	.....	.....	11.51	.....	.....	11.39	.....	.....	11.50
	2	26.62	.....	.....	.....	.....	.....	.....	26.67	.....	.....	26.66
	3	45.11	.....	.....	.....	.....	.....	.....	.....	.....	.....	44.68
H. B. McDonnell	1	10.25	.....	.....	11.39	.....	.....	.....	11.25	.....	.....	.....
	2	25.79	.....	.....	25.80	.....	.....	.....	26.02	.....	.....	.....
	3	44.14	.....	.....	.....	.....	.....	.....	44.31	.....	.....	.....
J. M Bartlett	1	.....	.....	.....	11.35	.....	.....	.....	.....	.....	.....	.....
	2	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....
	3	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....
L. H. Merrill	1	10.22	.....	.....	.....	11.37	.....	.....	.....	.....	.....	.....
	2	26.53	26.49	.....	.....	.....	.....	.....	.....	.....	.....	.....
	3	44.40	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....
F. S. Shiver	1	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	11.14
	2	26.38	.....	.....	.....	.....	.....	.....	.....	.....	.....	26.38
	3	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	44.08

[1] In these cases magnesium nitrate was used, but the solvent not given.

total phosphoric acid.

Sulph. acid and potassium nitrate, prec, cold, stood at 40° C.	Ignited with silica (Fries).	Solution in hydrochloric acid.	Aqua regia.	Ignited, and dissolved in nitric and hydrochloric acid.	Nitric and hydrochloric acid, 4 hours at 50° C.	Ignition and solution in hydrochloric acid.	Ignition and solution in aqua regia.	Fused with carbonate and nitrate of soda, solution in nitric and hydrochloric acids.	Boiled five hours with nitric and hydrochloric acid.	Sulph. acid and HgO (Huston).	Fused with carbonate of soda, solution in hydrochloric acid.	Sulph. acid and nitrate of soda, prec. at 65° C., stood 1 hour at 65° C.	Sulph. acid, prec, cold, stood 4 hours at 40° C.	Fused with carbonate of soda, silica removed, solution in HCl.
P. ct.	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*

Analyst.	Sample No.	Nitric and hydrochloric acid.	Hydrochloric acid and potassium chlorate.	Mg. nitrate, solution in nitric acid.	Mg. nitrate, solution in hydrochloric acid.	Mg. nitrate, solution in nitric and hydrochloric acid.	Nitric and hydrochloric acid, prec. at 40° C., stood at air temperature.	Aqua regia, prec. at 40° C., stood at air temperature.	Sulph. acid, Hg or HgO, and permanganate (De Roode).	Sulph. acid, HgO, permanganate and ferrous am. sulph, before precipitating.	Mg. nitrate, solution in nitric acid, silica removed.	Sulph. acid, and potassium or sodium nitrate (Burney).
		P. ct.	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*
N. W. Lord	1	.....	.....	.....	11.29	.....	.....	.....	.....	.....	.....	.....
	2	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....
	3	.....	44.35	.....	.....	.....	.....	.....	.....	.....	.....	.....
J. S. Merry	1	10.40	.....	.....	11.26	.....	.....	.....	.....	.....	.....	.....
	2	26.14	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....
	3	43.55	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....
H. E. Curtis	1	.....	10.13	.....	11.22	.....	.....	.....	.....	.....	.....	.....
	2	.....	26.24	.....	.....	.....	.....	.....	.....	.....	.....	.....
	3	.....	44.11	.....	.....	.....	.....	.....	.....	.....	.....	.....
A. M. Peter	1	.....	.....	.....	.....	.....	.....	.....	11.19	.....	.....	.....
	2	.....	.....	.....	.....	.....	.....	.....	26.15	.....	.....	.....
L. W. Wilkinson	1	.....	.....	.....	11.02	.....	.....	.....	.....	.....	.....	.....
	2	.....	.....	.....	26.40	.....	.....	.....	.....	.....	.....	.....
W. J. Gascoyne	1	.....	.....	.....	11.26	.....	.....	.....	.....	.....	.....	.....
	2	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....
	3	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....
W. L. Tibballs	1	.....	10.58	.....	11.47	.....	.....	.....	11.18	.....	.....	.....
P. H. Seymour	2	.....	26.80	.....	.....	.....	.....	.....	26.62	.....	.....	.....
	3	.....	45.16	.....	.....	.....	.....	.....	44.49	.....	.....	.....
B. W. Kilgore	1	.....	.....	.....	11.23	.....	.....	.....	.....	.....	.....	.....
	2	26.25	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....
	3	43.53[1]	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....
F. B. Carpenter	1	.....	.....	.....	11.17	.....	.....	.....	.....	.....	.....	.....
	2	26.23	.....	.....	.....	.....	.....	.....	25.33	.....	.....	.....
	3	43.76	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....
H. A. Huston	1	11.15	10.26	.....	11.32	.....	.....	.....	17.62	11.38	.....	11.20
Arthur Goss	2	26.48	26.69	.....	26.35	.....	.....	.....	26.28	26.39	.....	26.64
Dumont Lotz	3	43.29	45.06	.....	45.13	.....	.....	.....	56.09	45.38	.....	45.00

[1] Residue from this solution was fused, and gave 0.65 per cent additional.

total phosphoric acid—Continued.

Sulph. acid and potassium nitrate, prec. cold, stood at 40° C.	Ignited with silica (Fries).	Solution in hydrochloric acid.	Aqua regia.	Ignited, and dissolved in nitric and hydrochloric acid.	Nitric and hydrochloric acid, 4 hours at 50° C.	Ignition and solution in hydrochloric acid.	Ignition and solution in aqua regia.	Fused with carbonate and nitrate of soda, solution in nitric and hydrochloric acids.	Boiled 5 hours with nitric and hydrochloric acid.	Sulph. acid and HgO (Huston).	Fused with carbonate of soda, solution in hydrochloric acid.	Sulph. acid and nitrate of soda, prec. at 65° C., stood 1 hour at 65° C.	Sulph. acid, prec. cold, stood 4 hours at 40° C.	Fused with carbonate of soda, silica removed, solution in HCl.
P. ct.	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*
.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....
.....	.....	.....	.....	.....	.....	26. 16	.....	.....	.....	.....	.....	.....	.....	.....
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.....	.....	.....	.....	.....	.....	.....	11. 24	.....	.....	.....	.....	.....	.....	.....
.....	.....	.....	26. 51	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....
.....	.....	.....	44. 90	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....
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.....	.....	.....	.....	.....	.....	.....	.....	44. 25	43. 69	.....	.....	.....	.....	.....
.....	11. 55	.....	.....	.....	.....	.....	.....	.....	.....	11. 31	11. 35	.....	.....	11. 26
.....	26. 45	.....	.....	.....	.....	.....	.....	.....	.....	26. 51	26. 39	.....	....	26. 56
.....	42. 82	.....	.....	.....	.....	.....	.....	.....	.....	45. 17	44. 57	[2]44. 91	45. 16	44. 69

[2]The mean of two determinations from this solution precipitated on the usual 65° bath gave 44.85 per cent.

It will be noticed that the wet methods in use during the past year have proved entirely unsatisfactory for No. 1, which enforces the points already brought out in previous reports in regard to the necessity of not using the methods with nitric and hydrochloric acid, or hydrochloric acid and potassium chlorate, for goods containing phosphorus in cotton seed meal or similar material.

The table contains results by twenty-six different methods. Some of these differ but slightly from each other, yet I have thought it best to publish the results in detail.

Dr. De Roode called the attention of the members of the association to a method of determining both phosphoric acid and nitrogen in different portions of the same solution. The method is precisely the same as the first form of the Kjeldahl, but the solution after the addition of permanganate is made up to volume, and an aliquot part taken for the determination of phosphoric acid, and another portion for the determination of nitrogen. In my own laboratory this method was tried so far as relates to the phosphoric acid; the solution gave considerable trouble on account of a precipitate containing manganese and phosphoric acid, which appeared when the solution was neutralized, and was not wholly soluble in nitric acid. This accounts for the enormously high results given in the table of special work on phosphoric acid. The matter was easily corrected by adding to the neutralized solution a small quantity of ferrous ammonium sulphate. I prefer this to the addition of an organic substance like oxalic acid. I do not feel at liberty to recommend this method, although it has given good results, because it seemed that such a recommendation should only be made by the agreement of the reporters on nitrogen and on phosphoric acid. The method is recommended for trial during the coming year by vote of the association.

In regard to the statements of Messrs. Johnson and Osborne, relating to the precipitation of the phosphoric acid in solutions containing alumina, and stating that the results were more accurate by the original Sonnenschein method, Mr. Morse reports that he was unable to get concordant results on alumina goods before he took this matter into account. On the other hand, the work of Mr. McDonnell and the work in my own laboratory failed to confirm the observations of Messrs. Johnson and Osborne.

In my own laboratory it was found that the solutions resulting from treatment of phosphates with sulphuric acid sometimes contain crystals after standing for a day or more in the laboratory. These crystals were examined, and were found to contain no phosphoric acid, although other observers have reported that crystals were formed which contained phosphoric acid.

INSOLUBLE PHOSPHORIC ACID.

The chief interest in the work of the reporter on phosphoric acid seems to be associated with the citrate-soluble phosphoric acid, and hence considerable attention was given this matter. The work on the insoluble in No. 1 by the different workers is fairly satisfactory; that upon No. 2 is far from satisfactory, but this is probably due to the fact that comparatively few workers ever determine the citrate solids in this class of goods, and are hardly familiar with the manipulation involved in handling raw bone. It requires considerable practice to handle them readily and get concordant results, yet concordant results are possible on this class of goods, as I have repeatedly shown in my own laboratory work.

I think that the matter of the citrate soluble in raw bone is of more importance than would at first appear, as, especially in the central part of the country, large amounts of goods are sold, which consist of a mixture of about one-half raw bone and one-half steamed bone, which has sometimes had just enough acid added to make it necessary to determine the soluble phosphoric acid, and hence to class the goods with the superphosphates. The raw bone in this class of goods is seldom attacked at all by the acid used, and hence I deemed the investigation of the solvent power of the citrate solution upon raw bone to be a matter of considerable importance.

I do not believe that such material as No. 3, high-grade, aluminic phosphate, can be examined satisfactorily by the present method of working. The results among different workers vary very widely, and of course no one supposes that the method at present in use was ever intended for goods of this class.

Some special investigations upon the question of insoluble phosphoric acid in these samples was made by Messrs. Morse and McDonnell and myself. The results appear in the following table:

Table of results of special work on insoluble phosphoric acid.

Investigator.	Method.	No. 1.	No. 2.	No. 3.	Dissolved South Carolina rock.	Raw South Carolina rock, 27.67 per cent total.	Basic slag, 19.42 per cent total.	Raw rock, from bone fossilized with carbonate of lime. Total 31.02 per cent.
		Per ct	*Per ct.*	*Per ct.*	*Per ct*	*Per ct.*	*Per ct.*	*Per ct.*
F. W. Morse	Official	1.34	16.06	14.88	.....	.....	.....	.....
	Peterman's alkaline solution, 30 min., 65° C.	2.00	22.16	2.87	.....	.....	.....	.....
H. B. McDonnell; report by William Frear.	Official	1.27	17.96	14.50	1.28	.....	.....	.....
	Neutral citrate + 1cc., 10 per cent citric acid.	1.34	17.44	15.69	1.25	.....	.....	.....
	Neutral citrate + 1cc. 10 per cent ammonia.	1.39	18.62	12.64	1.28	.....	.....	.....
H. A. Huston	Official	1.26	17.57	19.61	.....	25.86	12.44	.....
	Neutral citrate made from C. P. citric acid.	1.31	16.72	18.47	.....	.....	.....	.....
	Official; solution made by Arthur Goss.	.....	.....	19.65	.....	.....	.....	.....
	Neutral citrate + 1 gr. citric acid.	1.26	12.87	26.31	.....	23.08	9.30	.....
	Neutral citrate + ammonia sp. gr., 0.85 equivalent to 1 gr. citric acid.	1.29	21.00	9.95	.....	26.93	13.93	.....
	Peterman's solution A, 30 min., 65° C.	1.16	22.31	2.33	.....	.....	.....	.....
	Peterman's solution B, 30 min., 65° C.	.....	.....	2.78	.....	.....	.....	.....
	Joulie's solution, 18 hours at air temperature.	.....	.....	1.13	.....	.....	.....	.....
	100cc. Ammonia sp. gr., 0.95, 30 min., 65° C.	.....	.....	33.20	.....	.....	.....	.....
	Pyrophosphate of soda, 50 gr. to liter, 30 min., 65° C.	.....	.....	33.59	.....	.....	[1]32.68 35.20	.....
George L. Teller	Official	1.73	.....	.....	.....	.....	.....	.....
	Official, with different citrate solution.	1.41	.....	.....	.....	.....	.....	.....

[1] Material caked badly in flasks.

In general it will be noted that, with the exception of the second determination by Mr. Morse on No. 1, the addition of considerable alkali or acid to the citrate solution has comparatively little effect; whereas it appears that upon the raw bone the acid solution is much more effective than the neutral, and that the presence of free alkali in the solution retards the solvent power of the solution to a very marked extent.

On the other hand, the results upon No. 3 show clearly that the alkaline solution dissolves far more than the neutral, and that the presence of free acid in the citrate solution retards the solvent power of the solution to a very considerable extent. In the work done by myself, the second set of determinations was made with a citrate solution made up from chemically pure citric acid. This was undertaken because of a letter received from Dr. Charles Gibson, of Chicago, who said he had obtained a much lower insoluble on this material by using chemically pure citric acid. The difference is not very great, but seems to be in favor of the statement made by Dr. Gibson. The two neutral solutions made, one by myself and the other by Mr. Goss, gave results that I think are extremely good, and show that the solutions used by us are at least alike whether they are neutral or not.

The next point investigated was the matter of the definite excess of acid or alkali in the solution, and here the results, especially upon No. 3, were very marked. The work upon Peterman's solution was made in order to have something to compare with the work done by Mr. Morse. The two solutions (A and B) were made at the same time, and made up as nearly alike as possible. The object in making two solutions was to see how nearly two strongly alkaline solutions could be made to act alike. In past years some criticism has been made upon work done by myself upon aluminum phosphate, and the suggestion offered that the solutions called neutral by me were really acid. I believe that the definite excess of acid and alkali used by me is sufficient in either case to make the solution distinctly acid or distinctly alkaline; but in order that there might be no doubt as to the alkalinity of the solution used, I omitted the citric acid entirely in one case, and used an amount of ammonia equivalent to that in Joulie's solution, but without any citric acid being present. This solution must certainly be admitted to have not been acid. The treatment which the aluminum phosphate is said to receive, would tend to convert it into pyrophosphate, and hence the attempt was made to treat it with some specific solvent for pyrophosphate, and for this purpose a pyrophosphate of soda was selected.

The result with Joulie's solution, and the low results invariably obtained by Peterman's alkaline solution, lead me to believe that practically all the phosphoric acid in this material is equally available.

Last year I reported some work which seemed to indicate that more phosphoric acid was dissolved by citrate solution where a large quantity of material was worked upon than where a small quantity was used, but I think the matter might be explained by the fact that the action of the solvent did not continue long enough to dissolve all that might otherwise have been dissolved. The work was therefore repeated, and, instead of thirty minutes, five hours' digestion at 65° C was used. The results were as follows: In each case 100 cc. of neutral citrate solution was used. When 0.5 gr. of aluminum phospate was taken, 3.69 insoluble was obtained; when 2 gr. were taken, 3.93 per cent of insoluble was found; when 8 gr. were taken, 5.60 per cent insoluble was found, showing that the results obtained last year were due to the fact that the action of the solvent was not complete at the time the operation was interrupted. In the table I have also included some work upon basic slag containing 19.42 per cent of phosphoric acid, a sample of ground South Carolina rock, prepared by myself from a museum specimen, containing 27.67 per cent of total phosphoric acid, and a sample of bone fossilized with carbonate of calcium, containing 31.02 per cent total. It will be noticed that the basic slag, when treated with the pyrophosphate, absorbed phosphoric acid from the pyrophosphate, as was to be expected.

To your reporter it seemed that the most important thing connected with the determination of the insoluble was a reliable method for rendering the citrate solution neutral. After considerable experimenting the following plan was devised, which has worked very satisfactorily in my laboratory, and the reports from a few other workers, to whom the method was sent, are such as lead to the belief that the method will be acceptable to most workers. The method of preparing the citrate solution is the same as usual, but when it is supposed to be approximately neutral, the citric acid in the solution is replaced by hydrochloric acid, and the final test of neutrality is really made in a dilute solution of ammonium chloride; the details of the process are as follows: Prepare a solution of fused calcium chloride, 200 gr. to the liter, and add four volumes of strong alcohol; neutralize the mixture, using a small amount of coralline as a preliminary indicator; test the neutrality finally by withdrawing a small portion, diluting with an equal volume of water, and testing with cochineal. 50 cc. of this solution will precipitate the citric acid from 10 cc. of the citrate. In testing the neutrality of the citrate solution, take 10 cc. of the approximately neutral citrate solution, add 50 cc. of the alcoholic solution; filter at once, using folded filter (about 35 or 40 cc. will run through); add an equal volume of water, and test the neutrality of the filtrate with neutral cochineal; when the neutral point is very nearly reached, larger quantities of the citrate solution, and a proportionately larger amount of the chloride of calcium solution may be used.

The change of indicators was made because of the fact that a strong solution of calcium chloride interferes with the indications furnished by cochineal, while a strong solution of amonium chloride does not give reliable results with coralline. In connection with the work on developing this method of neutralizing the citrate solution, citric acid, the strength of which had been accurately determined, was mixed with enough ammonia of known strength to exactly neutralize it. The solution so obtained was tested a number of times by this method, and found to be strictly neutral. Moreover, the addition of very minute quantities of either half normal acid or half normal alkali to this solution gave very marked results in the final test of neutrality. A solution of strictly neutral ammonium citrate, which was synthetically prepared, was of course much more dilute than the official solution, since it was impossible to accurately determine the strength of such strong ammonia as would necessarily be used in making an official citrate solution. This strictly neutral solution gave with coralline a color which was distinctly to the purple side.

The results upon the insoluble phosphoric acid differed so much among different workers that I asked a number of workers to send me portions of their citrate solution, which I tested by this method. The great differences in the amount of insoluble found could not be explained by the amount of acidity or alkalinity of the solutions. For example, the solution used by Mr. Farrington was very slightly toward the acid side, yet the result obtained by Mr. Farrington in No. 3 is the lowest in the entire series. The solution sent me from the Kentucky Station was decidedly toward the alkaline side, yet the result on insoluble in No. 3 is considerably above the average. The solutions used by Mr. Knorr, and by the Geneva Station, were as nearly as might be neutral, yet the results differed quite widely in this case. The solution used by myself was neutralized by this method. I believe the widely varying results upon the insoluble in No. 3, then, are not to be accounted for by the difference in the character of the solution, but are due to the fact that the digestion of the material with citrate solution is interrupted at a time when the solution is still going on with a considerable degree of rapidity.

The work reported by me last year would seem to indicate that the material continues to dissolve at a reasonably good rate for four hours, and that the solution is nearly complete at the end of five hours, yet the result at the end of ten hours showed that some material had gone into solution during the last five hours of the treatment.

Upon October 9 the solutions received from Mr. Farrington, Mr. Knorr, and Mr. Peters, of the Kentucky Station, and my own solutions, were again tested with the alcoholic chloride of calcium in order to see if any change had taken place during

the two months in which they had been standing in my laboratory. The solution sent by Mr. Farrington was found to be neutral, the solution by Mr. Peters was still slightly alkaline, and the solution made by myself was neutral, while the solution made by Mr. Knorr was decidedly acid. The acidity of the solution used by Mr. Knorr is readily accounted for by the fact that some mold had developed in this solution, and such common molds, it is well known, always render solutions in which they grow acid.

It is of interest to note that the solution of Mr. Farrington, which was formerly slightly acid, had become, as nearly as might be, neutral. The solution used by Mr. Farrington contained the coralline which he had used to test the neutrality with, yet this showed no change in color, and in fact seemed to have gotten into such a condition that the addition of 2 or 3 cc. of half normal acid or alkali to the solution gave no indication with this coralline of any change in the neutrality of the solution. The coloring material appeared to have become entirely inert to any small quantities of alkali or acid.

Mr. Arthur Goss did nearly all the preliminary work on the method of neutralizing the citrate solution by the use of chloride of calcium solution, and to his careful and extended work the perfection of the method is very largely due.

On washing the citrate insoluble of the phosphate of alumina with water, a considerable quantity of reddish-brown material is always carried through the filter, even when the first filtrate containing the citrate solution is clear. Some investigations on this point have led me to believe that when the phosphate of alumina of this character is treated with citrate of ammonia, a substance is formed, containing citric acid, iron, and possibly alumina, and phosphoric acid, which is insoluble in the citrate solution, even when diluted, but which is soluble in water. The following results will illustrate this: 2 grs. of the aluminum phosphate were treated as usual with the citrate solution, and filtered and washed five times with water; washings 6 to 10, inclusive, were collected, and found to contain 0.07 per cent of phosphoric acid; washings 11 to 15, inclusive, were collected, and found to contain 0.02 per cent of phosphoric acid. Another sample was treated with the citrate solution as usual, but washed with a dilute solution of the citrate solution; washings 6 to 10 contained 0.08 per cent of phosphoric acid; washings 11 to 15 were lost; after the fifteenth washing with dilute citrate solution, five washings were made with pure water, and these washings showed that 0.47 per cent of phosphoric acid had been removed by the water, even after the fifteen washings with the citrate solution. The washings with the dilute citric solution were invariably clear, while at once upon the renewal of the washings with water the peculiar reddish-brown material was dissolved, and the appearance was practically the same as occurred when water was used immediately after the separation of the original citrate solution with which the material had been digested.

All of these filtrates were evaporated to dryness and ignited with magnesium nitrate before determining the phosphoric acid contained in them. I consider that somewhat more accurate results may therefore be obtained by using the dilute cold solution of citrate of ammonia for washing this material.

I am indebted to Mr. S. L. Goodale for many interesting and valuable samples of phosphates, including many that contained alumina, and also preparations from this class of material that have properties which promise to be of importance in connection with the availability of the phosphoric acid in such material. I regret that the time at my disposal did not permit me to give more attention to this interesting question.

Mr. Jacob Reese also sent samples of basic slag in lumps and in powder, and also a very full description of the process of the production and preparation of it.

Mr. William F. Lewis writes in reference to acidulated phosphate of alumina, claiming that the aluminic sulphate produced is injurious to plants. Field experiments, and also experiments in the greenhouse, are now under way bearing on this point, but I can not at this date give the final results. The present indications are that the

results with the nonacidulated phosphate of alumina will be better from an agricultural standpoint than those with the same phosphate which has been acidulated. Experiments are also in progress using sulphate of alumina alone.

REMARKS BY DIFFERENT ANALYSTS.

GEORGE L. TELLER. The citrate solutions were made as nearly neutral as possible by using coralline as an indicator. This indicator has always been very unsatisfactory in my hands.

F. W. MORSE. Having had some experience with the Redonda phosphate, which I believe is the same as the keystone, perhaps you will allow me to say that the official method is worthless for such phosphates, in my estimation. Nitric acid will not leave a clear residue, while aqua regia (3 parts hydrochloric acid, 1 part nitric acid) acts readily with little heat, leaving a clear residue of silica.

My experience with molybdate solution corroborates Johnson and Osborne. Previous to seeing their note, I was unable to get any closely concordant results, but by following their method, or by letting stand over night, as in my report, I have no difficulty with the analyses.

B. H. HILL. No. 3, total. After prolonged boiling with 30 cc. concentrated nitric acid and a small quantity hydrochloric acid, there was still considerable residue left undissolved. This residue was washed thoroughly and fused with acid potassium sulphate, and an additional amount of phosphoric acid obtained from it.

HARRY SNYDER. No. 3. In filtering the solution of citrate soluble, much trouble was had in keeping the precipitate from being carried through mechanically; when paper was used for filtering, the particles were carried through in the filtrate; these separate upon standing a short time. When asbestus was used this trouble was avoided. The results given are with asbestus filter. The amount carried through when paper was used was found to be by determination 4.88 per cent, and 4.83 per cent of phosphoric acid when the filtrate was refiltered through asbestus.

F. S. SHIVER. In all cases sulphuric acid and potassium nitrate worked splendidly, with the exception of the last (No. 3), in which case there was a precipitate of ferric sulphate, which separated out and required heating to get back into the solution. For this reason I do not consider it a good method of treating for this particular material.

GENEVA EXPERIMENT STATION. No. 3 gives much trouble by going through filter, even the best filter paper made to hold barium sulphate, etc.

H. E. CURTIS (reported by A. M. PETER). The soluble was washed out on a filter pump, and the citrate of ammonia solution was neutralized by both litmus paper and coralline solution.

In regard to the latter point, our experience has always differed materially from what seems to have been that of most association chemists. Our practice is to throw into the solution at the same time a piece each of red and blue litmus paper, and when the two pieces, upon striking the liquid, approach as nearly as possible a common neutral shade, the neutralization is considered accomplished. The solution is also tested by adding a few drops of it to a drop of coralline solution in a small porcelain dish, and we find that the solution, neutralized as above, gives a bright crimson color with the coralline. Our experience has been, that while litmus paper and coralline, used in the manner described, agree in their indications, the litmus paper is decidedly the more delicate indicator, showing smaller changes in the solution than does the coralline. In my opinion, neither of these gives sufficiently sharp indications, and it would be highly desirable to have some more certain means of determining the neutrality of the citrate solution.

H. B. MCDONNELL. I found some time ago that an old solution of coralline that had been in the laboratory for two years was worthless, and am inclined to believe that the solution should not be relied upon after it has stood many weeks, say six or eight weeks.

THE DETERMINATION OF PHOSPHORIC ACID AND NITROGEN IN COMMERCIAL FERTILIZERS IN THE SAME WEIGHED QUANTITY OF SUBSTANCE.

By Rudolf de Roode.

Weigh 2.5 gr. of the fertilizer. Place in a Kjeldahl digestion flask, and proceed exactly as in the official Kjeldahl method, plain or modified, as the case may be. When the contents of the flask have cooled, add about 50 cc. of water, boil a few minutes to dissolve everything soluble, cool the flask in a current of cold water, transfer to a 250 cc. measuring flask, rinse digestion flask with successive small portions of water, make up to mark with rinsings, and mix. Filter through a dry, fluted filter into a dry flask.

Take 50 cc. corresponding to 0.5 gr. of fertilizer, nearly neutralize with ammonia, add 15 gr. of ammonium nitrate or its equivalent, and proceed exactly as in the official method for total phosphoric acid.

Take with a pipette a portion corresponding to 0.7 or 1.4 gr. of fertilizer, let run into a distillation flask, add potassium sulphide and caustic soda, and proceed as in the official Kjeldahl method.

To save the trouble of transferring, Kjeldahl digestion flasks may be selected of the proper capacity, and graduated with a mark at 250 cc. A pipette which will deliver 70 or 140 cc. may also be obtained, and Kjeldahl blanks corresponding to each may be made with pure sugar to test these quantities of the reagents.

Results with different methods of total P_2O_5 determination upon different materials obtained by J. A. Fries.

Method I.—HNO_3 + little HCl.
Method II.—Burney's, adding KNO_3 little by little.
Method III.—Ignited with Mg $(NO_3)_2$, then dissolved as in I.
Method IV.—Ignited with SiO_2, then dissolved as in I. (1.5—2 gr. SiO_2 to 2 gr. of fertilizer.)

Materials.	I.	II.	III.	IV.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*
Cotton-seed meal	0.97	3.90	3.77	3.95
Bran (wheat)	.73	3.89	3.87	3.98
Ground fish	4.20	4.43	4.26	4.43
Dried blood	.80	.83	.81	.87
Raw bone	10.66	10.66	10.27	10.65
Fish guano	12.07	12.19	12.18	12.39
Mona guano	21.91	22.21	22.14	22.27
Guarantee guano	*10.33	10.04	9.82	9.82

* Contained Al_2O_3, while precipitate by method IV contained none.

The above results are means of two or more determinations.

NOTES UPON RESULTS.

In case of aluminum phosphate, the favorable influence of slight alkalinity, and the unfavorable influence (though less marked) of slight acidity, of the citrate upon the solubility of the phosphate, as noted last year by Gibson, are corroborated.

Johnson and Osborne's results upon the influence of temperature in precipitation of goods rich in Al_2O_3 are not confirmed.

WITH REFERENCE TO DETERMINATIONS OF TOTAL P_2O_5.

Mr. Fries's results show that while the HNO_3 + little HCl effects a clear, and apparently perfect solution in all cases, in some cases not all the P_2O_5 is precipitated

from this solution by molybdate. He has observed this to be the case in the presence both of acid and of alkali albuminates which he has prepared. Especially is it true of the solution of vegetable materials, such as cotton-seed meal and bran, and, to a less degree, of those containing fish, while the phosphoric acid of blood and bone are completely precipitated. Dr. Dancy's conclusions respecting the insufficiency of this method in the case of goods containing cotton-seed meal are fully corroborated by Mr. Fries's results.

The method of previous ignition with magnesium nitrate is not shown to be fully adequate either. In addition to the danger of loss by intumescence, the substance often fuses to a hard mass, adhering strongly to the sides of the vessel. Mr. Fries has further noted that in several instances he found the fused mass, after apparently complete ignition, did not completely remain in solution in the acid after boiling, but settled out in flocculent or gelatinous precipitates, which upon examination were found to contain magnesium and phosphoric acid.

On the other hand, Mr. Fries observed no fusion on ignition of any of the substances named after intimate mixture with pure, finely pulverized SiO_2, even when a high temperature was maintained for some time. No intumescence occurred, a short time was sufficient for complete ignition, the residues were completely soluble without precipitation, and the molybdate precipitations were usually more complete than in solutions made otherwise, and never less complete, thus affording no evidence of the formation at any temperature likely to be used, of any difficultly soluble pyrophosphates. In the single case of the guarantee guano, where the P_2O_5 found by this method was less than that obtained by direct acid solution and by Burney's method, the results were, nevertheless, identical with those obtained by ignition with $Mg(NO_3)_2$, and the precipitate by the direct acid solution method was found to contain Al_2O_3, which was absent from the precipitate by the SiO_2 method. This affords the explanation of the apparently low results by the latter method, although the reason for the different behavior of the Al_2O_3 in the two cases is unknown.

Burney's method requires more manipulation than the other methods, and sometimes deposits crystals after dilution to required volume that contain, according to Mr. Fries, small amounts of phosphoric acid even after careful washing. Moreover, unless the strongly acid solution be neutralized with ammonia, and then slightly acidified by HNO_3, the molybdate precipitation is incomplete.

In case of the committee samples it is observed that the results in case of No. 3 are altogether too low by this method, while even in case of No. 1 the results are lower than by the silica method, although Mr. Fries obtained quite concordant results by the two methods when working on cotton-seed meal alone.

As for the method proposed by Mr. de Roode, we have found it unreliable, owing to the formation of a brownish to purplish flocculent precipitate after ammonia has been added. This precipitate varies in amount with the quantity of permanganate used; it contains phosphoric acid, and is insoluble in an excess of acid, though apparently slightly soluble in an excess of ammonia. The results obtained by the two different analysts owe their wide divergence undoubtedly to the formation of more or less of this precipitate, since different quantities of permanganate were used in the different cases.

PAPER ON INSOLUBLE PHOSPHORIC ACID.

[Forwarded to reporter by F. W. Morse.]

The work described in the following lines was undertaken in connection with a series of experiments not yet completed, and it is reported now, not with the idea of proving anything, but with the hope that it may, in conjunction with the work of others, throw some light upon the vexing question of citrate soluble phosphoric acid.

It is, in brief, a comparison of the action of neutral and alkaline solutions of ammonium citrate upon several kinds of phosphatic material.

The alkaline solution used was that known as Peterman's, described in Bulletin 19, Division of Chemistry, Department of Agriculture, p. 31.

Its excess of ammonia over the neutral point was determined by titrating with standard hydrochloric acid, and found to be equivalent to 12.75 grs., or 64.5 cc. of ammonia of 0.91 sp. gr. per liter.

The neutral solution was prepared according to the official method prescribed by the A. O. A. C.

The estimations of the solubility of the phosphates in these two solutions were carried out under as nearly identical conditions as could well be done.

The bath was a circular vessel, 12 inches in diameter and 5 inches in depth, with a cover pierced with four holes large enough to allow the flasks to pass through and float upon the water. A thermometer was immersed in the center of the bath to the depth of 1½ inches.

In most of the trials a neutral and an alkaline solution were carried along together. The method of digestion, solution, and estimation of phosphoric acid was that prescribed in the official method for determination of citrate soluble phosphoric acid.

The materials used, together with the total phosphoric acid, and the amounts soluble in water and in neutral and alkaline ammonium citrate solutions, are given in the following table:

	Total P_2O_5.	P_2O_5 soluble in water.	P_2O_5 soluble in neut. am. cit.	P_2O_5 soluble in alk. am. cit.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*
Ground bone	26.65	.....	6.22	2.94
Do	26.45	.....	9.31	4.29
Mixed fertilizer	11.85	7.90	2.61	1.95
Thomas slag	21.25	.....	5.68	3.02
			5.32	3.08
Redonda phosphate	46.85	.....	21.91	45.51
			25.25	45.31
Keystone phosphate	44.97	.....	30.04	42.05

The two last are iron and aluminum phosphates, and contain no lime.

These results indicate that lime phosphates are not so readily soluble in alkaline ammonium citrate as in the neutral, while iron and aluminum phosphate are more soluble, in fact being almost completely soluble in the alkaline solution.

A sample of dissolved bone black containing 16.52 per cent of phosphoric acid, of which 14.23 was soluble in water, was digested without previous washing with water. The result was that 0.12 per cent was insoluble in neutral citrate, and 2.89 per cent in alkaline citrate solution, or, expressed in a different manner, the neutral solution dissolved 2.17 per cent and the alkaline solution .60 per cent; or, in other words, rendered insoluble, 0.48 per cent of the phosphoric acid, which had been soluble in water.

This surprising result has been reached too late for further work before the meeting of the convention, and I mention it tentatively, knowing that alkalies are favorable to the reversion of superphosphates.

SECTION 3.—RECOMMENDATIONS BY THE REPORTER.

(1) That in all cases where ignition is resorted to, hydrochloric acid be used as a solvent.

(2) That the following methods be added to those already in use for the determination of total phosphoric acid:

(*a*) Burney's method, using either nitrate of soda or potash, adding 2 to 4 grs. at the beginning of the operation, and a little when the digestion is nearly complete; or adding the nitrate in small amounts from time to time;

(*b*) Digestion with strong sulphuric acid and mercury or mercuric oxide, as in Kjeldahl method;

(*c*) Solution in 15 to 30 cc. strong hydrochloric acid and 5 to 10 cc. nitric acid. This is described in detail in the directions for the official methods in the appendix;

(*d*) The aliquot part of the solution taken for analysis shall be from 0.25 to 1 gr., according to the richness of the phosphate;

(*e*) After neutralizing the acid solution of the phosphate with ammonia, a few drops of nitric acid are to be added to render the solution clear.

(3) That the method of neutralizing the citrate solution by use of alcoholic solution of calcium chloride and cochineal, as described under the report on insoluble phosphoric, be adopted as an alternate method.

(4) That ammonic hydrate be used in making the citrate solution.

The PRESIDENT. It is now proper to present any papers bearing on this subject.

Mr. Wilkinson presented the following:

PROPOSED METHOD FOR THE ANALYSIS OF NATIVE PHOSPHATES CONTAINING IRON AND ALUMINUM.

The recent discovery in Florida of native phosphates containing iron and aluminum demands that the association adopt a method for the analysis of these phosphates, and the following one has been found to give satisfactory results, with the exception that the lime determined by this method appears to be a little too small, and the method known as the alcohol or Glaser method is thought to be more reliable for the estimation of CaO.

(a) *Estimation of total* P_2O_5.—Dissolve 3 grs. of the phosphate in 25 cc. of HCl and a little nitric, evaporate to dryness over a water bath, redissolve in con. HCl and hot water, and dilute to 300 cc. Filter out 50 cc. through a dry filter, and determine the total phosphoric acid as usual.

(b) *Estimation of iron and aluminum oxides.*—To the filtrate from the molybate precipitate obtained above add ammonium hydrate in slight excess, heat in water bath at 65° C. for about thirty minutes, filter, wash with hot water, dissolve the precipitate off the filter with HCl, reprecipitate with ammonia, filter, dry, ignite, and weigh as oxides of iron and aluminum.

(c) *Estimation of ferric oxide.*—Titrate with standard permanganate solution.

(d) *Estimation of lime.*—To the filtrate from iron and aluminum add a little ammonia, then ammonium oxalate in excess, and determine lime by the usual method.

Mr. Wilkinson also presented the following:

PROPOSED CHANGES IN THE ASSOCIATION METHOD FOR THE ESTIMATION OF "WATER SOLUBLE," AND "ACID" OR "INSOLUBLE," PHOSPHORIC ACID, AND THE ADOPTION BY THE ASSOCIATION OF A METHOD FOR THE ANALYSIS OF NATIVE PHOSPHATES CONTAINING IRON AND ALUMINUM.

(1) *Estimation of water soluble phosphoric acid.*—In the estimation of water soluble phosphoric acid it is proposed to obtain the solution by placing the sample directly on a filter over a 300 cc. graduate, and wash by filtration, allowing all of the water to pass through each time before adding more, instead of washing by decantation in a small beaker. This decreases the manipulation and gives higher results in all samples examined, especially in acid phosphates. In washing by decantation there appears to be dissolved a phosphate of lime which immediately precipitates, and when the supernatent liquid is poured off through a filter, this soluble phosphate is retained by the filter, whereas, when the sample is placed directly on the filter, this soluble phosphate passes through the filter, and is precipitated in the graduate, causing murkiness, which may be readily cleared up by the addition of a little nitric acid.

The results of three different samples are here given:

	Acid phosphate No. 1.	Acid phosphate No. 2.	Complete fertilizer.
	Per cent.	*Per cent.*	*Per cent.*
Association method	10.25	10.94	7.46
Proposed method	11.21	11.30	7.85

(2) *Estimation of "acid" or "insoluble" phosphoric acid.*—Treat 1 gr. of the fertilizer sample with 100 cc. of neutral ammonium citrate solution in the usual way, and make up the solution of "acid" soluble to 100 or 150 cc.; take 50 cc., and proceed to estimate the P_2O_5 by the association method. This has been found to give results equally as accurate in the three samples analyzed, as first removing the water soluble and then treating the residue with ammonium citrate. If the available phosphoric acid is all that is considered of any value to both manufacturer and consumer, why not estimate the "acid" soluble as above proposed, take it from the total phosphoric acid, leaving the total available, and do away with the estimation of water soluble? This will give justice to all parties concerned, and reduce both the labor and expense of the analysis one-third. The following concordant results of three different samples show the reliability of both methods under consideration.

	Acid phosphate.	Complete fertilizer No. 1.	Complete fertilizer No. 2.
	Per cent.	*Per cent.*	*Per cent.*
Association method	2.95	0.97	1.53
Proposed method	3.06	0.93	1.48

(3) *Washing the molybdate pre ipitate in the estimation of phosphoric acid.*—Washing with pure water, as recommended by the association at its last session, there is frequently a precipitate formed in the filtrate from the precipitate of phospho-molybdate of ammonia. This is avoided by acidifying the water with nitric acid.

The following paper was presented by the secretary:

THE DETERMINATION OF TOTAL PHOSPHORIC ACID IN FERTILIZERS CONTAINING ORGANIC MATERIALS.

By F. S. Shiver, Ph. G.

It will be noticed on p. 98 of Bulletin No. 24 Chem. Div. U. S. Dept. of Agr., that there is described by Dr. Burney a method for the determination of total phosphoric acid by treatment with sulphuric acid and potassium nitrate.

As stated there, the action of the nitric acid is secured not only at a high temperature, but in a nascent state; hence its applicability to substances containing large amounts of organic materials, which are with difficulty oxidizable.

It may be well here to describe this method as used by myself for the last two seasons of fertilizer work:

2 gr. of the substance are brought into a 200 cc. flask and treated with 10 cc. of concentrated sulphuric acid; after well mixing, the flask is placed on a sand bath and heated until gentle ebullition takes place; then a few crystals of potassium nitrate are added, and the addition is continued until the contents of the flask assume a white or pale straw color; the operation is then considered complete.

After cooling, water is carefully added and the contents of the flask made up to the mark; then 50 cc. are taken out for precipitation.

Ammonia is added in considerable excess, and nitric acid is added until the precipitate, caused by ammonia, ceases to appear. By so doing I have found that the addition of ammonium nitrate is unnecessary to secure thorough precipitation. Precipitation with molybdate, and digestion at 65° C. for an hour follow as in the usual way. I have found that the sulphuric acid and the sulphates introduced in no way interfere with the precipitation, which is complete in an hour.

I may be safe in saying that one out of every six fertilizers manipulated in this State is ammoniated with cotton-seed meal, and hence our desire to obtain a method which would give quick and accurate results in determining total phosphoric acid in fertilizers containing this material.

There is no necessity of my calling attention to the fact, which every chemist who has had to deal with fertilizers knows, that nitric and hydrochloric acids, and in fact all the wet methods used up to the present time, whether due to incomplete oxidation or to a condition which the phosphoric acid assumes in which it does not yield to the molybdate precipitant, do not give correct results, invariably being too low by from 0.3 to 0.5 per cent.

This has been the experience of every analytical chemist connected with fertilizer control stations, and is demonstrated by the fact that at last year's meeting of the association the magnesium nitrate method was specified as the one to be used where cotton-seed meal was present.

The magnesium nitrate method is, as shown by its results, an accurate one; but is it a rapid one? In practice I have found considerable difficulty with the method, owing to its liability to sputter, and therefore chance for error; the fact also that it requires so much time has been our reason for not using it in our official fertilizer work. I have compared the sulphuric acid and potassium nitrate methods with the magnesium nitrate method on fertilizers containing cotton-seed meal, and have found them to disagree by not more then 0.15 per cent, which is within the working limit of error, and in a great many instances the results were identical.

On comparison with the method of ignition and solution in acid, in which case we believe we obtain the true total contents of phosphoric acid, I have found that the two methods do not disagree by more than one-tenth per cent, which is within the working limit of error also.

The great advantage, however, which this method possesses over these two is the great saving of time. In very few instances have I failed to obtain a clear solution

in less than one-half hour; in some very refractory materials, however, it has taken me somewhat longer. The saving of time must be a question of the very first importance to those stations exercising fertilizer control, and especially those which have a large amount of this work to do, and a comparatively short time in which to accomplish it.

It should not be understood from what has been said that this method is confined entirely to fertilizers containing cotton-seed meal. I have used it with success on fertilizers containing fish scrap, dried blood, tankage, and other organic materials. In none of these instances have I failed to obtain a good solution and accurate results, but I am of the opinion that this work is best confined to fertilizers containing cotton-seed meal.

I have found that, on comparison with the nitric and hydrochloric acid methods, in fertilizers containing such materials as dried blood, fish scrap, tankage, and bone meal, the results are almost identical; although the nitric acid solutions are very highly colored, showing the incomplete oxidation of organic matter, there is not enough left to seriously interfere with the precipitation of the ammonium phosphomolybdate.

The sulphuric acid solutions of the same materials are always colorless or pale straw-color, showing the complete oxidation of all organic matter present. To the analyst there is certainly a great deal of satisfaction in this.

The results obtained on a fertilizer containing dried blood, by the nitric and hydrochloric acid method, and this method, did not differ by more than 0.05 per cent, and in some instances were identical. Compared with the magnesium nitrate method, results were 0.07 per cent apart; with potassium chlorate and hydrochloric acid, 0.12 per cent; with ignition and solution in acid, 0.08 per cent.

On a sample of bone meal analyzed some time ago I obtained by this method 26.38 per cent, and by nitric and hydrocholoric acids 26.38 per cent, the best solution being in favor of the former method. On a sample of complete fertilizer containing cotton-seed meal, I obtained by this method 11.14 per cent, by ignition and hydrochloric acid, 11.08 per cent, both being good solutions.

In giving these results for comparison I have used the averages of those obtained by others as well as by myself, and I think I am justifiable in accepting them as representing the true percentages of phosphoric acid in the samples.

It will be seen from these results that this method bears favorable comparison with others which have had long trial and given good results, and in instances where these methods have failed it has given results comparable with the only other rapid method capable of being used at all, viz, the magnesium nitrate method.

I do not care to be considered as urging this method as one to be adopted for all substances containing phosphoric acid, for there are some materials with which it does not give accurate results, notably with highly siliceous materials, unless proper precautions are taken to separate silica; with acid phosphates the tendency is to give high results; but neither of these classes of materials need be treated in this way, as either nitric or hydrochloric acid will answer the purpose.

The principal objection to this method is the formation of such a large quantity of finely divided calcium sulphate, which has a tendency to pass through the pores of the filter on being filtered, but this difficulty is by no means insurmountable.

This method is certainly one which affords quick and accurate results, and is worthy of at least more extended investigation.

I have made over 300 determinations with it, and on using others as a check I have found that it always gives close agreement with the methods now in official use.

Mr. ROBINSON. I think the method suggested by Mr. Wilkinson is not accurate. It has fallen to my lot to determine the alumina and iron in a good many of the Florida phosphates, and I have decided that the Glaser method is the only method to be depended upon. I have a paper

showing some determinations which have been published in my report in regard to the availability of Florida phosphates, and I will offer that as a paper, if it be considered desirable.

The PRESIDENT. As it is necessary to expedite matters in order to finish the business of the association to day, I suggest that the paper be held over until the consideration of the method to which Mr. Robinson has referred.

At 12:30 o'clock a recess was taken until 2 p. m.

AFTERNOON SESSION, FRIDAY.

The convention was called to order by the president at 2 p. m., and the recommendations of the reporter on phosphoric acid were declared to be next in order.

Mr. HUSTON'S first, second, and third recommendations were then read and adopted.

Mr. HUSTON (after reading his fourth recommendation). I hope that somebody will move to amend it so as to virtually include Mr. De Roode's method. I do not like to do so without agreement with Dr. Frear, because it is included in the matter of nitrogen as well as phosphoric acid.

Mr. DE ROODE. I move as an amendment, that my method be placed either between the methods for nitrogen and phosphoric acid, or after both, as an alternate method, to be used in case the fertilizers should contain both nitrogen and phosphoric acid. I have prepared this method for use only when nitrogen and phosphoric acid are both present. If this amendment should be adopted, I will write out a description of the method. I want to state that if a small excess of permanganate be added, as directed in case of the nitrogen determinations, and the color after this addition be permanent, this color can be made to entirely disappear by boiling for a few minutes. This can in no way interfere with the determination of phosphoric acid.

Mr. FREAR. I do not wish to see that adopted just yet. The results obtained in my laboratory with the permanganate indicate that it is hardly a thing to be relied upon for commercial work, work that can not be checked immediately, and so in my report upon nitrogen I did not feel safe in recommending the use of permanganate. I think this method should be more fully tested before it is adopted.

Mr. DE ROODE. I withdraw my amendment, and move to have this method referred to the reporters on phosphoric acid and nitrogen for the ensuing year.

Mr. VAN SLYKE. I suggest that "examination in case the method is modified for nitrates" be included in that.

The PRESIDENT. I can see no objection to including it, and put the motion that way.

Carried.

Mr. Huston's original recommendation was then adopted, as were also the fifth and sixth, without further discussion.

The seventh recommendation was then read, and a motion made that it be adopted.

Mr. COOKE. In regard to the question of the preparation of the ammonium citrate solution, it has been suggested that the solution might be made by finding out the quantities of ammonia and of citric acid equivalent to each other, to prepare this neutral solution, the supposition being that the solution that was neutral to litmus was the triammonium citrate. On exactly making up a solution of the triammonium citrate according to molecular weights, it was found that the triammonium citrate—what would be chemically the neutral citrate—was not neutral, but strongly alkaline, requiring between 5 and 6 per cent more of citric acid before it became neutral. I have tried these two solutions on some 25 different commercial fertilizers as they came into the laboratory in the course of analysis, and, as might have been expected, got higher results on insoluble with the chemically neutral—the triammonium—citrate, than with that neutral to litmus, averaging about 0.33 per cent more of insoluble phosphoric acid. I have determined the equivalent quantities of ammonia and citric acid as exactly as possible, and according to my figures it is 105.6 per cent of citric acid to 100 per cent of ammonia, as compared with what would be required by molecular weights. In other words, it takes 5.6 per cent more of citric acid than the theory would require. Whether or not this is exactly correct I do not know, but think that the method outlined by Mr. Huston would give a very good chance to find the exact figures on that question. It seems, to be the first feasible method of determining ammonium citrate known to be neutral, that has yet been presented to the association. It seems, too, that to determine the equivalent quantities, it would be much easier to make up a solution of ammonium citrate known to be right by taking ammonia and finding its strength by standard acid, and taking commercial citric acid and finding its strength by standard alkali, and then uniting these in proportions which would make the required neutral ammonium citrate. I have used this method with the figure given, 105.6, and found it to be a rapid and easy way of making up the standard ammonium citrate solution.

Mr. HUSTON. What indicator was used?

Mr. COOKE. Squibb's litmus.

Mr. HUSTON. I can not see how that could be successful. I have given that up, because I think it hopeless to try to hold the solution of ammonium hydrate together long enough in the strength necessary to make up the citrate solution. I have not dared to try to determine its strength, the ammonia required being so strong.

Mr. PAYNE. I desire to offer a recipe for the preparation of the citrate solution, as follows:

To prepare ammonium citrate solution: Take citric acid 1,800 grs., distilled water 6,000 cc. Dissolve the acid in the water, pour into a bottle, and add gradually (shaking at the same time) 22 per cent conc. ammonia, 1,500 cc. The solution should now be slightly acid (if it is not, make it so by addition of citric acid); add 10 per cent ammonia, drop by drop, until exactly neutral (testing with alcoholic solution of cochineal); dilute with water until the sp. gr. stands at 1.09 at 20° C. Make up in a 3-gallon demijohn or 2½-gallon bottle.

Rather new, much cheaper than the other methods, much quicker, and requires less skill.

Mr. LORD. I have made ammonium citrate on the same general plan, but have found it difficult, using coralline, to make two solutions that were precisely the same.

Mr. PAYNE. I propose to use cochineal instead of coralline.

Mr. HUSTON. It would be necessary to dilute the solution if cochineal were used to prevent bleaching.

The PRESIDENT. I desire to call attention to the fact that the question before the house is not a particular recipe for making ammonium citrate, but Mr. Huston's recommendation.

It was moved that the recommendation be adopted. Carried.

Mr. HUSTON. I move, in order to cover Mr. Payne's proposition, that after the words "carbonate of ammonia," the words "or ammonium hydrate" be inserted.

Mr. FREAR. I move as a substitute, that after the words "heat to expel the carbonic acid," the words "or neutralize directly with ammonium hydrate" be inserted. Carried.

Mr. PAYNE. I move that on page 228, Bulletin 28, section 4, after the words "water of about the temperature of 65° C.," the words "containing 25 per cent of citrate of ammonia solution," be inserted. I have found that this will expedite matters a good deal.

Mr. HUSTON. I have done some work on that, and find in certain cases it is good and in others not so good, and I suggest that the matter be referred to the reporter for the ensuing year.

Mr. VAN SLYKE. I move as an amendment that this matter be referred to the next year's reporter on phosphoric acid. Carried.

Mr. LORD. I have made some experiments on phosphates which came into my hands three or four years ago, to test the influence of prolonged washing, and have found that continuing the washing with water meant a steady reduction in the amount of insoluble acid found. I have since that time adopted an invariable rule in washing insoluble acid. I transfer the solution to the filter, according to the official directions, as rapidly as possible, and after letting this liquid run through, fill the funnel a definite number of times with water, letting the water run through after each addition, and make that number invariable. In this way I obtain very concordant results. I wish to ask whether that uniformity of washing is usually practiced?

No answer.

Mr. WILKINSON. I move that the following method for the determination of the water soluble phosphoric acid be adopted in place of the one given in the official methods:

Weigh 2 gr. Place directly on a 9 centimeter No. 589 Schleicher & Schüll filter; wash by filtration, allowing all of the water to pass through the filter each time before adding more, and continue the washing until the filtrate measures not less than 250 cc.

I wish to strike out all of the first sentence given in the official method, because I think if the sample be properly prepared that manipulation is entirely unnecessary.

Mr. BARTLETT. This matter has been considered two or three times at the meetings of this association, and considerable data, and the opinions of quite a number of good chemists can be obtained showing that it is better in this determination to put the fertilizer directly on the filter. I have made several comparisons of the different methods, and have always found that leaching on the filter will give higher results than any other way.

Mr. VAN SLYKE. I move as an amendment that the matter be referred to the reporter for the ensuing year.

Mr. FREAR. I am personally rather inclined to favor the direct washing on the filter, but know that good chemists differ decidedly on that point, and I think it would be wise to have the reporter take up this matter and test it, and I therefore second the motion that the question be referred to the reporter with the urgent request for attention during the next year.

Mr. MCDONNELL. I remember that this matter was brought up two years ago, and the same motion made, that it be referred to the reporter to be considered another year. I would like to see the method suggested by Mr. Wilkinson adopted, as I think the results would be more uniform.

The PRESIDENT. I am satisfied that these requests and suggestions to the reporter will receive more attention during the ensuing year, because the secretary has kindly offered to send to each new reporter a complete list of all the matters referred to him.

Mr. HUSTON. I believe that in very many cases there would be no practical difference in the results by the proposed method, but I hesitate to vote for its adoption on account of the relatively large size of the hole in the sieve in use. There are many cases of superphosphates, particularly bone goods, with which I have to deal largely, in which I am convinced that all the soluble phosphoric acid could not be obtained without breaking up in a beaker.

Mr. PAYNE. I am satisfied that a large majority of the fertilizers in the United States are worked by the proposed process, and in the interest of uniformity I think the association ought to adopt it.

Mr. KILGORE. I have found that rubbing makes considerable difference, because chemists do not agree on what constitutes a homogeneous paste. I have made determinations in connection with another chemist,

and we have obtained different results. Then we took the same samples and made the determinations together, without any practical difference, which proved that the differences before obtained had been due to the rubbing.

Mr. LORD. I agree entirely with regard to the rubbing. I have obtained different results from those of an eastern chemist working on the same goods. After we had duplicated our results without agreement, we experimented on the subject of trituration, and I found that by rubbing the sample of the eastern chemists long enough, I could get exactly the same results obtained by him. I think that any action taken to change the method given should either do away with rubbing entirely, or retain the beaker. There should be no possibility of going back to extensive grinding.

Mr. VAN SLYKE. I think that the discussion has shown that some are of the opinion that rubbing makes a difference, and others think it does not. The only way to settle the question is to refer it to the reporter.

The amendment was then put and lost by a majority of one, and Mr. Wilkinson's original motion was adopted.

Mr. WILKINSON. I move that on page 229, line 4, the sentence commencing with the word "Digest" be made to read "Digest at about 65° C. for one hour; filter and wash with water acidified with nitric acid, or containing ammonium nitrate."

Mr. PAYNE. I think the method is satisfactory as it stands.

Motion put and lost.

Mr. WILLIAMS. I notice that Mr. Huston has not recommended any change in the temperature, and while this meets my view, it leaves the old trouble of not getting concordant results. I think this might be remedied by a slight modification in regard to time. According to the official method the flask was to be put in a bath at 65° C. and held there for thirty minutes, but nothing was said about the length of time required for the citrate of ammonium solution in the flask to reach 65° C. Some claimed that it required only three or four minutes, while others asserted that it required as high as twelve or thirteen minutes.

Mr. DAVIDSON. I move that in the case of nonacidulated goods, in determining the citrate insoluble, the time be changed from thirty to forty minutes from the time of insertion in the bath, the temperature of the bath being 65° C.

Mr. FREAR. I suggest that as long as the shape and size of the flasks are different, the same inequality will be present, even if this change be adopted. I can see no good reason for the change. The right direction would be to have a uniform proportion between the vessels and the baths.

Mr. GAINES. I think it would be inadvisable to have one method for the analysis of straight phosphates and another method for acidulated phosphates. I hope the change will not be made.

Mr. BARTLETT. I understand the gentleman's idea to be that the

phosphate should be treated at 65° C. for just thirty minutes. I think the only way to accomplish that, as long as flasks of different sizes and different thicknesses are used, is to take the temperature of the liquid in the flask.

Mr. VAN SLYKE. I move, as an amendment, that the matter be referred to the reporter on phosphoric acid. Carried.

Mr. Kedzie presented the following report:

REPORT ON SOIL ANALYSIS.

By R. C. KEDZIE.

In the following report little claim is made to originality, either of methods or of arrangement. The reporter has availed himself freely of assistance from all quarters, without attempting to designate the sources of information in each case, but would gratefully acknowledge the help he has received from the Agricultural Chemical Analysis of Dr. Caldwell; the pamphlet on Principles and Methods of Soil Analysis, by Dr. Richards, of the Department of Agriculture; the writings and contributions of Dr. Hilgard, of California; Crook's Select Methods; Sutton's Volumetric Analysis; Johnson's, Fresenius', and Frankland's Agricultural Chemical Analysis.

While the general principles involved in the analysis of soils and of ashes are the same, it is yet convenient to consider them separately, even though some repetition of details becomes necessary.

The question of the value and importance of analysis of the soil does not come before the reporter on methods of soil analysis. It is only his duty to recommend methods to be used by the Association of Official Agricultural Chemists.

The surface soil is the portion usually submitted to analysis, commonly spoken of as "the soil," and embraces that portion moved and disturbed by processes of tillage, and does not usually extend more than 9 inches from the top of the ground. The subsoil may be considered the second layer of 9 inches or more. If analysis is required of both soil and subsoil, these soils should be carefully separated from each other, and separately analyzed. The method of analysis will be essentially the same in each case.[1]

Mr. PATRICK. It is a very important matter, it seems to me, to fix upon the size of the sieve with which to obtain our fine earths. I came down to Washington, more than half, to find out about that sieve. The doctor has fixed it to be a sieve of 25 meshes to the inch. If this is satisfactory to everyone it will be satisfactory to me; I should be inclined to prefer, though, a specification of the orifice, because the wire of the screen varies a little, and if we could fix upon 1 mm. I should like it.

Mr. KEDZIE. I do not object to that. I adopted this because I had the sieve. I do not object to saying a sieve with an orifice of 1 mm., though I do not like the circular orifice. Sometimes we want to rub the materials through and it does not run through a round hole.

Mr. PATRICK. Will that amendment be acceptable?

Mr. KEDZIE. I would not object to that at all.

The PRESIDENT. I would call attention of the association to the

[1] Following this Mr. Kedzie submitted a scheme for analysis which, with a few amendments, was adopted as the official method. It will be found with Methods of Analysis, p. 233.—H. W. W.

fact that we have a sieve prescribed for fodders with a circular orifice 1 mm. in diameter. Wouldn't that be a good sieve to use?

Mr. HARRINGTON. In this connection I would like to say, as a great many of us in making these soil analyses make also mechanical analyses in which the sieves are specified with circular openings, it might be well to adopt one of those sieves, as we necessarily have them in the mechanical processes anyway.

Mr. PATRICK. Dr. Osborne has a 1 mm. sieve. I move that the association adopt for this purpose a sieve with circular openings 1 mm. in diameter, unless Dr. Kedzie has some objection to that.

The PRESIDENT. It is moved and seconded that the official sieve for the analysis of soils be one with circular holes 1 mm. in diameter.

Mr. FREAR. May I call attention to section 1, p. 228, Bulletin 28, the method for the determination of phosphoric acid? It mentions a sieve with circular perforations one twenty-fifth of an inch in diameter.

Mr. MCDONNELL. I think that is practically 1 mm. I think it would be better to have it specified as 1 mm., but it is essentially the same thing.

The motion was put and carried.

The PRESIDENT. Are there any further recommendations? I would ask Dr. Kedzie if many reports have been handed in on analysis of samples?

Mr. KEDZIE. Only two or three. The material for the analyses was sent out late. I had almost given up the idea of sending out any, but some of the chemists wrote and asked for samples, and I sent them out. As to the samples sent out not enough reports have been handed in to make any comparison whatever. Some analyses were made according to the methods prescribed here, and some by older methods.

Mr. VAN SLYKE. I move that the report as amended be adopted. Carried.

The president called for reports of committees.

Mr. Frear, for the committee on the amendment of section 3 of the Lindo-Gladding method for potash, submitted the following, which was adopted.[1]

Mr. PAYNE. I move that the consideration of miscellaneous business be discontinued, and the reports of work on fertilizer analyses be taken up as the order of business. Carried.

Mr. Frear presented the following report:

REPORT ON NITROGEN METHODS.

By WILLIAM FREAR.

ABSTRACT OF LITERATURE FOR THE YEAR 1890–'91.

As an introduction to this year's work of the association on nitrogen methods, your reporter has prepared a series of abstracts of the more important papers on this sub-

[1] See Methods for Analysis of Potash, p. 187.

ject so far as they have come to his notice. The more salient points may be indicated as follows:

The German Verband der landwirthschaftlichen Versuchs-Stationen, [42][1] adopted the following agreement for 1890–'91: (1) Nitrogen in blood, flesh-meal and similar organic substances is to be determined by the Kjeldahl or soda-lime methods. (2) Nitrate nitrogen in mixtures by Schloesing's, Grandeau's, or Lunge's methods; total nitrogen in such mixtures by the Kjeldahl, Jodlbauer, or simil armethods. Peruvian guano, raw or rectified, is included in this class. (3) Total nitrogen in nitrates by some direct method. (4) Total nitrogen in commercial ammonium salts by distillation with sodium hydrate.

Kebler [20] has issued a very full bibliography on the determination of nitrogen.

For the standardization of acid Hart and Croasdale [16] proposed sulphuric acid electrolytically prepared from pure copper sulphate, while Heidenhain [17] very strongly presses the merits of acid potassium tartrate.

Zaloziecki [46] brings to notice as an indicator for alkalimetric work a napthol benzöin which is said to be exceptionally sensitive to carbonic acid.

On the older methods little has been written except upon improvements in apparatus and reagents.

Guglielmo [15], Neesen [29], Stuhl [37], and Wells [45] have proposed various modifications of the mercury pump.

Hentschel [18] proposes a convenient form of apparatus for use with the hypobromite method.

Buchan [7] refers to the fusibility of the ordinary soda-lime as used in the Ruffle method, and to the attendant difficulty of cleansing the iron tube frequently used in this method, and describes a method of preparing a difficultly fusible soda-lime for such use.

With reference to the soda-lime method, Freydl [14] states that the low results in the analysis of guanidine by this method are due to partial oxidation of the ammonia formed, and to the formation of a little cyanic acid.

Berthelot [5] proposes the combustion of bodies poor in nitrogen in a current of hydrogen. This has, however, been tried before without any marked improvement in results.

Devarda [9] calls attention to the fact that the estimation of the ammonia produced during combustion, by direct precipitation by platinic chloride, leads to too high results, owing to the simultaneous precipitation of organic decomposition products of high molecular weight.

A great deal has been written upon the Kjeldahl method.

Lunge [26] and Moritz [27] have discussed the practicability of purifying the sulphuric acid for this method by the use of potassium nitrate. Link [24] reports that the desired result is not attained by boiling the acid with sulphur.

Concerning the accuracy of the Kjeldahl method in the analysis of substances free from nitrates, Argutinsky [1] finds that the Wilfarth modification gives excellent results, using metallic mercury; Devarda [9] maintains its accuracy to be equal to that by the soda-lime process. Proskauer and Zülzer [31] obtained excellent results in sanitary analyses, as did Müller [28], in soil analyses, after making slight modifications. Snyder [35] found that all the nitrogen of theine was obtained by this method. Argutinsky [1] states that with mercury no permanganate is required in the Wilfarth method, while Kebler [21] urges that both mercury (or its oxide), and permanganate are essential to the practical utility of the plain Kjeldahl process.

Proskauer and Zülzer [31] have obtained good results by using copper oxide or sulphate as a partial or an entire substitute for mercuric oxide.

The same writers state that they found no benefit arising from the use of sugar, benzoic acid, etc., in the case of substances rich in nitrogen, while Müller [28] recommends their use in the case of soils.

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[1] The bracketed numbers indicate the abstract referred to.

Argutinsky [1] states that mercurammonium compounds are freely decomposed by zinc dust, as used in distillation, and states that when it is so used the previous precipitation by potassium sulphide is unnecessary; but Kebler [21] says that the use of zinc is unnecessary if the proper burner be used in distilling, and Ulsch [41] states that the use of finely divided zinc is objectionable, although he assigns no reason. Boyer [6], however, states that zinc acts on ammonia in the presence of an excess of sodium hydrate to form an insoluble compound, and recommends the use of magnesium hydrate as the alkali where zinc necessarily occurs in the liquid to be distilled.

Niebling [30] suggests that as most of the ammonia is driven over early in the distillation, that the liquid be very gently heated at first. Argutinsky [1] claims that this operation should last fully forty-five minutes.

Kjeldahl [22] refers to the error arising from the carrying over of soda in the vapor, urges the necessity of washing the vapor thoroughly before its entrance into the condenser, and proposes a rather complicated form of apparatus for the purpose. A similar apparatus is proposed by Stein and Schwarz [36]; but Niebling [30] objects to the complicated character of both forms of apparatus, and urges that a simple bulb tube answers every need.

Foerster [12], Kebler [21], and Stutzer [38] all speak of the loss arising from the discharge of the exit tube of the condenser above the surface of the acid in an open beaker.

Proskauer and Zülzer [31] speak of congo red, rosolic acid, and cochineal, as well as methyl-orange, as fitted for use as indicators with this method, while Foerster [12] condemns the first three of these, as well as litmus, tumeric, and phenol-phthalein, but recommends methyl-orange, and especially a mixture of lacmoid and malachite green.

A number of communications have appeared touching upon the modifications of the Kjeldahl method for the analysis of nitrates. Ulsch [41] states that while zinc alone in sulphuric acid effects some reduction, it is attended by the formation of the lower oxides of nitrogen and consequent loss. Aubin and Quenot [3] have obtained good results by preliminary reduction with ferrous sulphate and subsequent treatment by the ordinary Kjeldahl process. They also obtained satisfactory results by the Jodlbauer modification, as also did Proskauer and Zülzer [31]. The latter writers state that loss of nitrogen by volatilization of ammonium sulphate can occur only when the walls of the flask are overheated. Foerster [12], comparing the Jodlbauer and Scovell modifications, decides in favor of the latter, because it gave in general somewhat better results, and because, in particular, he found a loss of nitrogen in the presence of chlorides when phenol was employed, but not when salicylic acid was used. Süllwald [39], it may be remarked, attacked Foerster upon this point, claiming that he has found no loss attending the use of phenol under the conditions indicated, and objects to the use of the salicylic acid; that it is almost impossible to effect the solution of sodium nitrate in sulpho-salicylic acid. Foerster [13], however, replied, reaffirming the loss of nitrogen previously reported, and states that the solution of the sodium nitrate in the salicylic acid, while admittedly difficult, may be secured by frequent vigorous agitation. The same investigator also compares the relative merits of zinc and sodium hyposulphite as reducing agents in both the Jodlbauer and the Scovell processes, and prefers the latter, because on the addition which may be made in one charge, there is cooling rather than heating, and therefore not the same danger of loss of nitrogen, and because no difficultly soluble salt is formed to interfere with the speed of reaction, and the objectionable presence of zinc salts in the later stage of the process is avoided. The writer enters quite fully into the theory of the several reactions involved.

Aubin and Quenot [3] suggest the precipitation of albuminoid nitrogen in mixtures by the use of tannin, and the separate determination of nitrates and ammonium salts in the filtrate, as well as of the nitrogen in the residue upon the filter.

Probably the most striking feature of the literature of this subject during the past year has been the activity manifested in the proposal of new methods for the determination of nitrates. Only the briefest mention can be made in this place of the various communications bearing upon this branch of the subject. Scheiding [32] has suggested a very convenient nitrometer. Cochius and Moeller [8] state that frequently failure in the use of Schloesing's (Schulze-Tiemann) method are due to want of proper proportion between size of apparatus and quantities of substance and reagents used, and they state the conditions under which they have attained the best results. Arnaud [2] proposes to determine nitrates in plants by the direct precipitation of the nitric acid as cinchonamine nitrate. Foerster [11] states that he has obtained accurate results in the analysis of sodium nitrate in the absence of magnesium chloride, by driving off the nitric acid by hydrochloric acid, and determining the loss. v. Koninck and Nihoul [23] decompose the nitrate by vapor of hydrochloric acid, conduct the chlorine formed in the mutual decomposition into potassium iodide, and estimate the liberated iodine by sodium hyposulphite. The majority of the methods proposed depend, however, upon the reduction of the nitrate by one means or another. Stutzer [38] effects the reduction by the use of aluminum (free from sodium alloy) in alkaline solution; Schmitt [33] by the action of iron and zinc in acetic acid, though Hollemann [19] states that he can not get good results by Schmitt's method; Boyer [6] by action of zinc and hydrochloric acid; Ulsch [41] by the use of reduced iron and sulphuric acid, or in another modification [40] by the use of coppered iron wire, while Vortmann [43] accomplishes the same result by electrolysis in the presence of a metallic salt, preferably a copper salt; though Becker [4], who ascribes the reduction to a metallic hydride formed by the electrolysis, rather than to the direct influence of the current itself, suggests that palladium hydride would probably be more quickly effectual.

In Stutzer's, Schmitt's, Boyer's, one of Ulsch's and one of Vortmann's methods, the ammonia found is distilled off and estimated by titration; in Ulsch's modification, in which coppered wire is used, the amount of ammonia is estimated by the hydrogen consumed in reducing the nitrate, as shown by the deficiency in the amount of that gas obtained from a known volume of standard acid in the presence of an excess of metal. Schulze had previously proposed to determine the deficiency of hydrogen obtained from a known weight of metal, but that is more tedious. Vortmann, in one of his modifications, performed the electrolysis in presence of an excess of copper sulphate, which is completely electrolyzed, and the excess of acid determined by titration with standard ammonia.

Several other methods have been proposed that are worthy of mention. Wagner [44] proposes to convert organic nitrogen into nitrates by treating with alkaline permanganate under pressure, and subsequent determination of nitrates by Eder's method. He does not find the method very satisfactory. Donath [10] has suggested that this excessive conversion of organic nitrogen into nitrites and nitrates by the action of hot alkaline permanganate may be utilized as a qualitative test for nitrogen, the nitrites formed being detected by diphenylamine. Wagner also proposes the determination of the nitrogen in nonvolatile nitro-derivatives and ethereal nitrates, by treatment with the alkaline permanganate in an open vessel, subsequent reduction by appropriate reagents, and estimation of the ammonia formed.

Smith [34] has made an elaborate study of the products of the action of brominated permanganate, formed by the action of permanganate in acid solution upon an alkaline bromide, upon ammonium salts, cyanides, urea, and albuminoids. He finds that this nitrogen is liberated as free nitrogen or converted into nitric acid, and suggests the feasibility of estimating the nitrogen in these and similar compounds by determining the excess of oxygen required for their oxidation by the reagent first mentioned, as compared with that consumed when permanganate alone is used in acid solution, although he has not worked out the method.

Loof [25] states that sodium salicylate is a good qualitative reagent for nitrates,

used just as ferrous sulphate is, and that the reaction is so delicate that it can be made roughly quantitative by colorinmetric comparison.

No attempt has been made to keep track of the special literature of water and urine analysis.

ABSTRACTS OF PAPERS ON QUALITATIVE AND QUANTITATIVE ESTIMATION OF NITROGEN, 1890–'91.

[Arranged alphabetically by authors.]

(1) ARGUTINSKY, P. *Notes on Wilfarth's modification of the Kjeldahl method.*—Excellent results were obtained by the use of C. P. sulphuric acid to which 200 gr. of phosphoric acid per liter had been added, also using metallic mercury, precipitating with 12 cc. of concentrated solution of potassium sulphide, distilling and titrating with $\frac{N}{10}$ sodium hydrate and cochineal. The use of permanganate was found unnecessary; neither is it needful to use the potassium sulphide, since the zinc dust completely liberates all ammonia held in any mercurammonium compounds. To completely distill the ammonia 45 minutes were requisite. Arch. Physiol., 1890, 46, 881; Chem. Zeit., 1890, 14, 41; Vtjsschr. Chem. Nahr., 1890, 102; Journ. Chem. Soc., 1891, 59, A, 362.

(2) ARNAUD. *Estimation of nitrates in plants by cinchonamine sulphate.*—Treat the sirupy aqueous extract (made by extracting the triturated plant with boiling water) with 40 per cent alcohol; evaporate the alcohol, successively remove chlorine by a little normal lead acetate, and any excess of lead by a few drops of sodium sulphate. Filter, reduce to very small volume by evaporation, slightly acidulate with acetic acid, boil, and mix with a warm solution of cinchonamine sulphate; the crystalline nitrate is at once precipitated. Keep in a cool place for twelve hours; filter, wash with cold aqueous solution of cinchonamine sulphate, then with very little cold water, dry, and weigh. Ann. Chim. Phys. [6], 19, 91-131; Journ. Chem. Soc., 1891, 59, A, 362–3.

(3) AUBIN, E., AND J. QUENOT. *Modification of Kjeldahl's method for the separate determination of nitrogen in nitrates, ammonium salts and organic compounds.*—In the presence of nitrates, the Jodlbauer modification gave good results. Reduction by use of ferrous sulphate and subsequent use of the plain Kjeldahl process gave good result on total nitrogen. For determination of albuminoid nitrogen, exhaust 1 gr. of the fertilizer with 30–40 cc. of a 2 per cent tannin solution (where ammonia-magnesium phosphate is present, digest for fifteen hours with 0.5 gr. tannin in 150 cc. of carbonated water); the nitrogen on the filter is classed as albuminoid, that in the filtrate as ammoniacal, after deduction of nitrates, which are separately determined by Schloesing's method. Bull. Soc. Chim. [3], 3, 322–6; Journ. Chem. Soc., 1890, 58, A. 921.

(4) BECKER, ADOLF. *Notes on Vortmann's electrolytic method for estimation of nitrates.*—The reduction is due to the metallic hydride deposited, for nitric acid alone is not decomposed by the current but only after the introduction of a metallic salt. Now, neither copper nor hydrogen alone reduce nitric acid; therefore the effect is to be attributed to their compound Cu_xH_x. It is very probable that palladium hydride would complete this reduction in a moment, since the speed of reaction must depend upon the nature of the hydride, and the circumstances influencing its production. Chem. Zeit., 1890, 14, 1557; Vtjsschr. Chem. Nahr., 1890, 5, 527.

(5) BERTHELOT. *Use of a current of hydrogen in the soda-lime method.*—The use of a current of hydrogen is recommended in the analysis of bodies poor in nitrogen. [The editor of Vtjsschr. Chem. Nahr., remarks that this was proposed twenty years ago by J. Lehmann, but shown by Mærcker and Arbesser (Zeitschr. Anal. Chem., 1873, 12, 449) to be useless.] Bull. Soc. Chem. [3], 4, 480; Chem. Zeit., 1890, 14, 306; Vtjsschr. Chem. Nahr., 1890, 5,526.

(6) BOYER, E. *Estimation of nitrates by reduction with zinc.*—The reduction of nitric acid by means of zinc and hydrochloric acid is usually incomplete, and attended by

loss of free nitrogen and its lower oxides. By strict adherence to the following method, however, accurate results are obtained: 5 gr. of granulated zinc, the size of peas, is put into a test tube 300 mm. long and 20 mm. in diameter; 10 cc. of a solution containing not more than 0.5 gr. of the nitrate is then introduced, and 5 cc. of hydrochloric acid (sp. gr. not less than 1.19) added. When hydrogen ceases to be evolved, add 5 cc. more of acid. In ten minutes the reduction is complete. Estimate ammonia by distillation as usual, but liberate it by use of magnesia instead of sodium or potassium hydrate, since the latter conduce, in the presence of zinc, to the formation of an insoluble ammonium derivative; mix with sodium or potassium hydrate until just alkaline, and then add 2 gr. of magnesia. Comp. Rend., 110, 954–6; Journ. Chem. Soc., 1890, 58, A. 1025; Ber. Deut. Chem. Ges., 1890, R, 437.

(7) Buchan, Alexander. *Preparation of soda-lime for Ruffle's method.*—The ordinary soda-lime melts in the iron tubes used for Ruffle's method, and renders it difficult to clean then. A soda-lime not melting at the temperatures attained may be prepared by forcing equal parts of fresh-burnt lime and crystallized sodium carbonate through a sixteen-mesh sieve, mixing in an iron pot and heating over an ordinary fire. Chem. News, 61, 231; Journ. Anal. and Applied Chem., 4, 326; Journ. Chem. Soc., 1890, 58, A. 1024, Ber. Deut. Chem. Ges., 1890, R, 753.

(8) Cochius, F., and Moeller, T. *Notes on Schloesing's (Schulze-Tiemann) method.*—Low results obtained in the analysis of explosives are attributed to excess of water, and disproportion between size of apparatus and quantity of material ordinarily used. With a 350 cc. flask, a 150 cc. measuring tube, 5–15 cc. ferric chloride (seven parts of the chloride to ten parts of water), 10–30 cc. of hydrochloric acid (35 per cent), and 0.3–0.4 grs. of potassium nitrate, boiling just enough to expel all the air, and using 25–30 cc. of water, the operation was complete in thirty to forty minutes, with a mean result of 13.81 per cent. But, if 80–150 cc. of water was used, the operation required seventy to ninety minutes, with a mean result of only 13.21 per cent. Chem. Zeit., 14, 33; Journ. Chem. Soc., 1891, 59, A. 107.

(9) Devarda, A. *Error introduced by the use of platinic chloride for the estimation of ammonia produced by the soda-lime method.*—Polansky and Latschenberger [Ztschr. f. Veterinärkunde, 1889, 2] having claimed that the Kjeldahl method gave too low results as compared with the soda-lime, the author attributes this to the erroneously high results obtained by the first-named investigators in determining the ammonia produced by the soda-lime process by means of platinic chloride. This salt forms insoluble compounds with incompletely decomposed nitrogenous organic compounds; the platinum compounds thus formed add to the weight in large proportion. Devarda found that the quantity of ammonia yielded by distilling the acid liquid of the soda-lime method, with an excess of sodium hydrate, exactly equaled in amount that indicated by direct titration of the same liquid and that obtained by the Kjeldahl method. Chem. Zeit., 14, 3; Zeitschr. Anal. Chem., 30, 360–1.

(10) Donath, E. *Test for nitrogen in organic substances.*—Heat the compound, in the presence of an excess of alkali, with alkaline permanganate, and nitrites and nitrates are successively formed. [Chem. Zeit., 1890, 14, 157.] The diphenylamine test is then used to detect any nitrite that may be formed. Zeitschr. Angew. Chem., 1890, 249; Monatsh. Chem., 1890, 15; Vtjsschr. Chem. Nahr, 1890, 233; Zeitschr. Anal. Chem., 1890, 457–8.

(11) Foerster, O. *Determination of nitric acid in Chili saltpeter by means of hydrochloric acid.*—Fuse 2–3 grs. of the salt in a weighed porcelain crucible with lid; after cooling add 25 cc. of hydrochloric acid (3 parts acid, 1 part water), and evaporate to dryness; repeat the latter process twice to expel all nitric acid. Heat the final residue for some time at 150° C., and then to dull redness. The loss equals the difference between NO_3 lost and Cl gained, and is to the N in the NO_3 expelled as 1 : .52828. This method is not applicable in the presence of magnesium chloride, which loses HCl in drying. Chem. Zeit., 14, 509; Ber. Deut. Chem. Ges., 1890, R, 667; Journ. Anal. and Applied Chem., 4, 457; Journ. Chem. Soc., 1891, 59, A. 107.

(12) By the same author: *Application of the Kjeldahl method to the determination of nitrates, alone and mixed with organic substances.*—The author, after sketching the development of the Jodlbauer and Scovell modifications of the Kjeldahl process, details experiments upon the above methods without the use of reducing agents, and with zinc dust and sodium hyposulphite as reducing agents. Without reducing agents, a loss of 0.37—.62 per cent of the nitrates present occurs; but, if heated until the phenol or salicylic acid begins to decompose, there is further combination of nitric acid and, at the same time, some volatilization. Sulphuric acid containing 5 per cent of phenol is better than that containing 10 per cent.

When zinc dust was used as a reducing agent, it was found, as had been noted by earlier investigators, that the use of platinic chloride to aid oxidation, proposed by Jodlbauer, led to a loss of nitrogen, and should be omitted. Zinc dust must be added in small quantities to avoid overheating, with attendant loss; some urge cooling at this point. Other sources of failure are the destructive action of gypsum, the formation of the difficultly soluble anhydrous zinc sulphate, which prolongs the operation, and the formation of zinc hydrate, which prevents quiet distillation, and requires the use of a large excess of sodium hydrate for its solution.

The hyposulphite is used as follows: After complete solution of 1 gr. of sodium nitrate in 30 cc. of sulphuric acid containing 6 per cent of phenol, 3–5 grs. of pure crystalized sodium hyposulphite was added, and, after the consequent reaction had ceased, 0.5 grs. of metallic mercury, and 20 cc. of sulphuric acid for every 30 cc. of the acid mixture. The oxidation was complete in 1½ hours without the addition of phosphoric acid or potassium permanganate. Results obtained with potassium nitrate gave 99.27–100.51 per cent of the nitrogen present, the mean of twenty-one determinations being 99.66 per cent. Hyposulphite has the advantage over zinc dust of cooling rather than heating the acid when it is added; of forming no difficultly soluble salt, but rather, one that hastens oxidation; the process is therefore more rapid than with zinc dust, and requires no other oxidizing reagents; further no precipitate, except that of mercuric sulphide, is formed, and distillation is more quiet.

Scovell's method, using, however, sulphuric acid containing 6 per cent of salicylic acid, gave better results with the hyposulphite than were obtained with phenol; 99.47–100.29 per cent of the total nitrogen was obtained, and a mean of 99.89 per cent in nine determinations.

Applied to determination of nitrogen in mixed nitric and organic compounds, hyposulphite acted equally well; and here, too, salicylic acid gave better results than phenol. Somewhat more unmixed sulphuric acid is required, and there is a greater heating at the time of solution, rendering a little cooling desirable.

In the presence of chlorides the use of phenol is attended with loss of nitrogen, probably as chlorpicrin. This may be avoided by precipitating chlorine by use of 10 cc. of a 0.6 per cent solution of silver sulphate. The use of mercurous salts for this purpose would often be inadvisable because mercurous phosphate forms an insoluble precipitate, which carries down and retains some of the nitrogen, thus causing loss. The use of salicylic acid in the presence of chlorides is not attended by loss.

Some loss was noted when the distillate was received in open vessels, and a large bulbed U tube was recommended for the purpose.

As an indicator, litmus, rosolic acid, turmeric, cochineal, and congo red, as well as phenol-phthalein are condemned, but methyl-orange, and especially a mixture of commercial lacmoid, 80 parts, extracted with 1,000 parts of 20 per cent alcohol, and malachite green, 13 parts, are recommended.

The author offers the following theory of these processes: The nitrates are reduced to nitrites, and nitro-sulphuric acid is then formed, when the nitro-group is given up to the phenol or salicylic acid. This reaction is hastened by zinc dust and by hyposulphite, although the reaction is probably too rapid to be explained by the action of the nascent hydrogen generated when zinc is used. The principal use of zinc is

the formation of amides by the generated hydrogen; in the case of the hyposulphite no such stage in the process is necessary, since this reagent forms ammonia directly from the aromatic nitro-compound; it is, indeed, possible that zinc dust may form hyposulphurous acid by the action upon sulphuric acid under these conditions, and in that way bring about the results attained. The nitro-phenols formed are probably mono and dinitro-compounds, for picric acid does not appear to be formed. Landw. Vers. Stat., 38 165–194; Journ. Chem. Soc., 1890, 58, A. 1466; Zeitschr. Anal. Chem., 1891, 30, 353–5.

(13) *Further notes on above topics.*—In reply to Süllwald, the author reaffirms the loss of nitrogen in Jodlbaner's method whenever chlorides are present. He also states that the solution of sodium nitrate in sulpho-salicylic acid is much hastened by frequent agitation. Chem. Zeit., 15, 76; Zeitschr. Anal. Chem., 1891, 30, 356.

(14) Freydl, F. *Oxidation of ammonia in the soda-lime method.*—The author finds the deficiency in nitrogen in the analysis of guanidine and biguanidine compounds by the soda-lime method to be mainly due to partial oxidation of the ammonia formed. (Cf. Makris, Annalen, 184, 371.) The presence of small quantities of cyanic acid in the soda-lime at the end of the operation accounts for a slight loss. Chem. Monatsh, 11, 120–8; Journ. Chem. Soc., 1890, 58, A. 1194; Zeitschr. Anal. Chem., 330, 359; Ber. Deut. Chem. Ges., 1890, R. 471–2.

(15) Guglielmo, G. *Modification of Spreugel pump.*—The author proposes a modification for more thorough deäeration of the mercury introduced. Rend. Acad. Lincei, 6, 2, 153–5; Journ. Chem. Soc., 1891, A. 524.

(16) Hart, E., and S. Croasdale. *Standardization of acid by means of sulphuric acid prepared by electrolysis of copper sulphate.*—The residual acid from C. P. copper sulphate, 5 gr. or less, is calculated from the weight of copper deposited on a platinum electrode by the formula: Sulphuric acid, $= \frac{97.82}{63.18}$ copper. This acid is then titrated by $\frac{N}{10}$ alkali. According to results obtained 1 cc. of $\frac{N}{10}$ soda $= .004767$ to $.004819$ gr. of sulphuric acid, or a mean of .004801 in seven determinations. Journ. Anal. and Applied Chem., 4, 424–7.

(17) Heidenhain, H. *Standardization of acid by potassium hydrogen tartrate.*—This salt is recommended because it crystallizes water free, is only very slightly hygroscopic, is readily pulverized, may safely be dried at rather high temperatures, has a high molecular weight, can be kept without change for a long time, and gives a sharpened reaction with phenol-phthalein. (It has been remarked that Bornträger made the same suggestion earlier.) Pharm. Rundschau, 1890, 133; Chem. Zeit., 14 R., 224, 1206; Zeitschr. Anal. Chem., 1891, 226.

(18) Hentschel, W. *Modification of apparatus for hypobromite method.*—See description *loc. cit.* Ber. Deut. Chem. Ges., 23, 2402–3; Journ. Chem. Soc., 1890, 58, A, 1341–2.

(19) Hollemann, A. F. *Schmitt's method for nitrates.*—Unable to get the satisfactory results secured by Schmitt. Chem. Zeit. 15, 77; Zeitschr. Anal. Chem., 1891, 30, 357.

(20) Kebler, Lyman F. *Bibliography of nitrogen determination.*—Journ. Anal. and Applied Chem., 5, 260–278.

(21) By the same author. *Notes on estimation of nitric nitrogen by Kjeldahl method.*—Upon the slow addition of acid, a slight but appreciable loss of nitrogen occurs. Permanganate and mercury in the form of either oxide or metal, are essential to the practical utility of the method. In distillation there is no need to use zinc if a Fletcher circular burner be used; the discharge tube of the condenser must dip under the acid. Journ. Anal. and Applied Chem., 1891, 5, 257–9.

(22) Kjeldahl, J. *Notes on the distillation in his method.*—The results are erroneously raised in distillation owing to solution of the glass, when a glass condenser is used, by action of the hot distillate; also by the carrying over of sodium hydrate in the steam. The first difficulty is obviated by the use of block tin for the worm and all downward conducting parts; the second, by washing the steam before passing it over.

For the latter purpose the author proposes a special apparatus, with the statement that a long, upright column, even when frequently bent, failed to keep back all the soda. Meddelelser fra Carlsberg Laboratoriet, 2, 197; Zeitschr. Anal. Chem., 1890, 461.

(23) v. Koninck and A. Nihoul. *Determination of nitric acid by decomposition with hydrochloric acid.*—The hydrochloric acid may be made to act upon the nitric acid with the formation of NO_3 and Cl; the latter gas is led into a solution of potassium iodide, and the liberated iodine determined by $\frac{N}{100}$ hyposulphite. Accurate results were obtained. Zeitschr. Angew. Chem., 1890, 477; Vtjsschr. Chem. Nahr., 1890, 381; Ber. Deut. Chem. Ges., 1890, 23, R. 705; Journ. Chem. Soc., 1891, A, 618.

(24) Link, Alb. *Removal of oxides of nitrogen from sulphuric acid.*—Almost no C. P. sulphuric acid is found in trade. Writer was unable to remove the nitrogen oxides by boiling the acid with sulphur. Pharm. Zeit. 1890, 35, 653; Chem. Zeit., 1890, 14, 288; Vtjsschr. Chem. Nahr., 1890, 52–3.

(25) Loof, G. *Sodium salicylate as a quantitative reagent for nitrates.*—Add 1 centigram of sodium salicylate to 5 cc. of aqueous solution of the substance to be tested; pour down the sides of the tube, so as to avoid mixing, 10 cc. of colorless, concentrated sulphuric acid. On slightly mixing the two layers, a deep red to pale yellow coloration occurs, varying so closely with the quantity of nitrate present as to admit a roughly quantitative colorimetric test by comparison with solutions of known strength. Pharm. Centr.-H., 31, 706; Ber. Deut. Chem. Ges., 1890, R, 776.

(26) Lunge, G. *Use of potassium nitrite for purification of sulphuric acid.*—The author urges that the use of potassium nitrite, as carried out by Meldola and Moritz, frees sulphuric acid from nitrogen only when used in exactly right proportions; if an excess be employed nitrous acid remains. Zeitschr. Angew. Chem., 1890, 447–8; Journ. Chem. Soc., 1891, A, 617.

(27) Moritz, E. R. *Ib.*—Cites experiments to overcome Lunge's objections against Meldola and Moritz's method of freeing sulphuric acid from ammonia by the use of potassium nitrite. Journ. Soc. Chem. Ind., 9, 443–4; Ber. Deut. Chem. Ges., 1891, R, 166.

(28) Müller, J. A. *Use of the Kjeldahl method in soil analysis.*—Soils treated 12–15 minutes with fuming sulphuric acid, to which a little benzoic acid had been added, yielded as much nitrogen as was obtained by the soda-lime process. Ann. Chim. Phys. [4] 22, 393-401; Ber. Deut. Chem. Ges., 1891, 24, R, 372.

(29) Neesen, F. *Modification of mercury pump.*—The author describes a modification of mercury pump constructed without the use of cocks. (See *loc. cit.*) Zeitschr. Instrumentenkunde, 2, 285; Zeitschr. Anal. Chem., 1890, 29, 441.

(30) Niebling, R. *Distillation in Kjeldahl's method.*—The author considers Kjeldahl's apparatus for the washing of the vapor (*q. v.*) as too complicated, and asserts that the use of a simple bulb tube is sufficient to keep back all soda. To avoid bumping he employs zinc dust, and recommends slow boiling in the early stage of distillation, because most of the ammonia passes over at this time. Chem. Zeit., 13, 1670; Zeitschr. Anal. Chem., 1890, 462.

(31) Proskauer, B., and M. Zülzer. *Notes on the Kjeldahl method as applied to hygienic analysis.*—The Kjeldahl-Wilfarth acid mixture is more rapidly oxidized with the aid of metallic oxides with substances rich in nitrogen, as foods. To 20 cc. of the mixture add 0.5 gr. of water-free copper sulphate or oxide, and 1 gr. of mercury. If there is little nitrogen present, the mercury is not needed in addition to the copper. The use of permanganate to complete oxidation is always desirable. Additions of benzoic acid, sugar, etc., are unnecessary in the case of foods, and retard action. With abundant nitrates the Jodlbauer modification is commended. In waste waters of low nitrogen content the use of zinc dust alone is sufficient for complete reduction, but nitrates must always be separately determined, or reduced in the appropriate manner. Loss of nitrogen may occur from heating the acid and substance too high just after mixture. No volatilization of ammonium sulphate occurs unless the walls of the flask are overheated. For titration either $\frac{N}{10}$ or $\frac{N}{100}$ soda may be used, with either Congo

red, rosolic acid, cochineal, or methyl-orange as an indicator. Zeitschr. Hyg., 1890, 7.186–222; Chem. Centr.-Bltt., 1890, 61, 142; Vtjsschr. Chem. Nahr., 1390, 103; Zeitschr. Anal. Chem., 1890, 460; Deutsche Chem. Ztg. 4, 377.

(32) SCHEIDING, F. *Apparatus for determining nitric nitrogen as nitric oxide.*—This apparatus is peculiar in the globular reservoir into which the measuring tube expands at the top, only the narrow lower portion being jacketed; and in the entrance of the soda and gas delivery tubes into the measuring tubes, the connecting piece being attached by rubber, if preferred, instead of being melted in. Measurements are taken at room temperature, and over soda instead of water. Chem. Zeit., 14 635–7; Journ. Chem. Soc., 1891, 59, A, 107–9; Zeitschr. Anal. Chem., 30, 357–9.

(33) SCHMITT, T. F. *Estimation of nitrates by the reducing action of acetic acid and metals.*—Into a 750 cc. Erlenmeyer flask 40 cc. of acetic acid and 15 gr. of a mixture of equal parts of zinc dust and iron filings are introduced; after shaking, add 50 cc. of a solution of the nitrate (equal to 0.5 gr. of the nitrate), mix thoroughly, and let stand 15 minutes, then add 15 gr. more of the mixed metals, and 30 cc. of water to prevent solidification. Reduction is complete in 30–40 minutes, and distillation may follow as in the Kjeldahl process. The temperature in the flask during the reduction should not exceed 75–80° C, since the ammonium acetate may decompose. Before distillation add enough soda to redissolve any zinc hydrate formed. The results obtained are accurate. Chem. Zeit., 14, 1410; Journ. Anal. and Applied Chem., 4, 455; Zeitschr. Anal. Chem., 1891, 30, 356–7

(34) SMITH, J. H. *Determination of nitrogen by brominated permanganate in acid solution.*—The author presents results of an elaborate study of the effects of an acid solution of permanganate to which an alkaline bromide is added, upon ammonium salts, cyanides urea, and albuminoids, showing that the nitrogen is more or less fully set free or oxidized to nitric acid, and determining the specific nature of the product in each case, and the condition under which such transformation is most complete. The author states that no non-nitrogenous organic substance known to him is more highly oxidized by brominated permanganate in acid solution, than by the acid permanganate alone; and that, therefore, any excess observed in the former case must be attributed to the oxygen consumed by the nitrogen. This fact is taken as the basis for a proposed quantitative method for nitrogen which the author has not yet fully worked out. The author seems to establish the fact that in the presence of chlorine some of the nitrogen in a solution treated with acid permanganate is converted into nitric acid and escapes estimation, rendering methods involving this combination unreliable. Chem. Zeit., 1890, 14, 1223–9; Zeitschr. Anal. Chem., 1890, 460; Ber. Deut. Chem. Ges., 1890, 23, R, 709.

(35) SNYDER, H. *Applicability of the Kjeldahl method to alkaloid analysis.*—Working with dilute solutions of theine, the average of 3 analyses gave 28.79 per cent of nitrogen instead of 28.86 per cent calculated, thus showing complete ammonification of the alkaloid. Journ. Anal. and Applied Chem., 4, 443–5.

(36) STEIN and SCHWARZ. *Distilling apparatus for Kjeldahl's process.*—The writers propose an apparatus similar to Kjeldahl's (*q. v.*) for washing the vapor to free it from soda. Zeitschr. Anal. Chem., 28, 420.

(37) STUHL, M. *Automatic mercury pump.*—The mercury is lifted to the reservoir by water pressure acting indirectly through compressed air, and regulated by a shifting weight. Ber. Deut. Chem. Ges., 24, 147–159; Journ. Chem. Soc., 1891, A, 640.

(38) STUTZER, A. *Estimation of nitrates by aluminum.*—The aluminum used should be prepared in other ways than by sodium, since by the latter method sodium alloys are generally present as impurities. Into an Erlenmeyer flask introduce 50 cc. of nitrate solution (equal to 0.5 gr. of potassium nitrate), dilute to 150 cc., add 25 cc. of sodium hydrate (density 32° Baumé); mix well, put in 2 to 3 gr. of aluminum foil, attach to the condenser, let stand over night, and then distill. The exit tube of the condenser should dip under the acid. Zeitschr. Angew. Chem., 1890, 695; Journ. Anal. and Applied Chem., 1890, 225; Journ. Chem. Soc., 1891, A, 617; Ber. Deut. Chem. Ges., 1891, A, 96.

(39) Süllwald, A. *Notes on Foerster's hyposulphite method.*—The writer observes that this method has an advantage over the Jodlbauer in requiring no cooling, and in relatively quick decomposition; but it has the disadvantage, where salicylic acid is used, that sodium nitrate is often incompletely dissolved in the sulpho-salicylic acid even after twenty hours, with agitation from time to time. Further, he finds no loss of nitrogen attending the use of Jodlbauer's method in the presence of chlorides. Chem. Zeit., 1890, 14, 1673 and 1748; 15, 149; Vtjschr. Chem. Nahr, 1890, 5, 526–7; Zeitschr. Anal. Chem., 1891, 30, 356.

(40) Ulsch, Karl. *Determination of nitric nitrogen by the deficit of hydrogen consumed in reduction (modification of Schulze's method).*—Schulze's method, by determination of the deficit of hydrogen formed in the presence of a weighed quantity of aluminum, is too slow, and zinc will not answer as a substitute. If the Schulze process be hastened, the reduction of nitrates is not complete. Ulsch substitutes an excess of coppered iron filings, free from oxide, and determines the deficit in the hydrogen obtained from a measured volume of $\frac{N}{10}$ sulphuric acid by comparison with a blank. Coppered iron may be made from 3 gr. of powdered iron and 20 cc. of a 10 per cent solution of copper sulphate. Displace the excess of liquid from the generator in which the operation has been performed, by air-pressure, produced by lifting the reservoir of the connected azotometer. Run in the sulphuric acid, and fill the azotometer with hydrogen; remove the excess of acid liquid as before, rinse with two portions (5 cc.) of $\frac{N}{10}$ sulphuric acid; finally bring water level in the azotometer to 0 at normal pressure, introduce the nitrate dissolved in 20 cc. $\frac{N}{10}$ sulphuric acid (not more than 10 mg. of the nitrate) into the generator; shake, heat one minute at 75° C., shake, heat again for one minute at 75° C., and then shake till bubbling ceases, and measure volume of gas with usual precautions. A deficit of 1.106 cc. of hydrogen equals 1 mg. of potassium nitrate.

The coppered iron can be used for 25 determinations, if made continuously. Platinized iron acts too quickly to completely reduce the nitrates. A heat higher than 75° C. yields an excess of hydrogen, and good results are obtained at 60° C., but more slowly; at 75° C. the reduction is complete in three minutes. Hydrochloric acid gives varying results; therefore an excess of chlorides, sufficient to replace all the sulphuric acid, will interfere with the accuracy of results. Zeitschr. Anal. Chem., 1891, 31, 182–195; Chem. Centr.-Blt., 1890, 2, 926–927; Journ. Chem. Soc., 1891, A, 617.

(41) By the same author. *Estimation by reduction with iron.*—Although zinc in acid solution converts nitric acid into ammonia, it is attended by the formation of lower oxides of nitrogen, and consequent loss, while the presence of the finely divided zinc during the distillation of ammonia is objectionable. Instead of zinc the writer recommends reduced iron free from nitrogen. Into a 250 cc. Erlenmeyer flask introduce 25 cc. of the nitrate solution (equal to 0.5 gr. of potassium nitrate) with 5 gr. of iron, and add 10 cc. of sulphuric acid (sp. gr. 1.35); shake; set on the water bath; insert a rubber stopper, through which a dropping bulb filled with water projects; boil five minutes; let cool; fill the bulb again with water; boil three minutes and cool, when action should be complete; then add 25 cc. of water and an excess of sodium hydrate, and distill off the ammonia. Iron wire and pulverized iron contain nitrogen, while well-prepared and well-preserved reduced iron does not. The basic oxide formed by the action of the sulphuric acid itself aids in the reduction. Hydrochloric acid is not a fit substitute for the sulphuric acid, and the presence of chlorides may interfere, though the writer has no data on that point. The end of the reaction is shown by the failure of a drop of the mixture to show a red color when treated on a porcelain plate with a drop of concentrated sulphuric acid. Zeitschr. Anal. Chem., 1891, 30, 175–182.

(42) Verband der landwirthschaftlichen Versuchs-Stationen im deutschen Reiche. *Official nitrogen methods for fertilizers,* 1890–'91.—At the Bremen

meeting, September 18, 19, 1890, the following methods were adopted: (1) Nitrogen in blood, flesh-meal, and similar organic substances, by the Kjeldahl or soda-lime methods. (2) Nitrate nitrogen in mixtures by Schloesing's, Graudeau's, or Lunge's methods; total nitrogen in such mixtures by Kjeldahl-Jodbauer, or similar methods. Peruvian guano, raw or rectified, is to be analyzed by this class of methods. (3) Total nitrogen in nitrates by a direct method. (4) Total nitrogen in commercial ammonium salts by distillation with sodium hydrate. Landw. Vers.-Stat., 38, 303, *seq.*; Zeitschr. Anal. Chem., 1891, 253, 254.

(43) VORTMANN, G. *Electrolytic estimation of nitric nitrogen.*—The method depends upon the fact that, although a current may be passed for several days through a solution of a pure alkaline salt, acidulated with sulphuric acid, without the formation of ammonia, if another metallic salt that is decomposed by the current be present, ammonia is formed. Copper sulphate is recommended for this purpose, although platinum and mercury salts as well cause a complete reduction; in the presence of zinc salts the nitric acid is not so certainly reduced. The solution of the nitrate is placed in a platinum dish with the requisite quantity of pure crystallized copper sulphate and electrolyzed, preferably with a current giving 1–2 cc. of oxyhydrogen per minute; when the copper is completely electrolyzed evaporate the liquid to a small residue, and distill off the ammonia as usual. The quantity of copper sulphate varies with the amount of nitric acid present; in the case of potassium nitrate, use 1 part of copper sulphate to 2 parts of the nitrate; a smaller proportion may be used, but then the copper must be deposited very slowly; and with a strong current even a large quantity of copper does not suffice.

A modification of the method is proposed, in which a carefully weighed quantity of the sulphate is used, and a measured quantity of $\frac{N}{5}$ sulphuric acid. The latter must be in quantity sufficient to hold in solution all the metals present; if the liquid should become alkaline from a deficiency of acid used, and a metallic oxyhydrate be deposited in consequence, more acid must be added at once, or ammonia will be lost by volatilization. Upon the complete deposition of the copper the excess of acid is determined by titration with $\frac{N}{5}$ ammonia, and the nitrogen determined by difference. In this modification, instead of using copper sulphate, the positive electrode may be coated with copper and introduced into the acidulated solution, and the operation completed by the transfer of the copper to the opposite electrode. If, however, too little copper be used, the direction of the current will change, and the copper be carried back to its starting point. These latter modifications are applicable in all cases where neutral nitrates are to be analyzed, and where no other salts are present that are permanently decomposed by the current. It must further be remembered that only half of the acid neutralized is held in combination by the ammonia, since the latter forms double salts, as illustrated by the following equation:

$$KNO_3 + 4H_2 + H_2SO_4 = KNH_4SO_4 + 3H_2O.$$

Tests of potassium nitrate by the first modification gave 13.64 to 13.87 per cent of nitrogen; mean of 4 analyses, 13.78 (theory, 13.88); by second modification, 13.60 to 14.14 per cent; mean of 6 analyses, 13.88 per cent. Ber. Deut. Chem. Ges., 1890, 2798–2801; Journ. Chem. Soc., 1890, 58, A, 1467; Vtjsschr. Chem. Nahr., 1890, 5, 527.

(44) WAGNER, R. L. *Estimation of organic nitrogen by alkaline permanganate.*—Heat 0.5–1 gr. of substance in a sealed tube with a capillary exit, for oxygen with 25–30 grs. of $K_2Mn_2O_8$ and 5 cc. of 25 per cent potassium hydrate at 150–170° C. for two and one-half hours. Then turn into a basin, reduce excess of manganate by manganous sulphate and sodium carbonate, and determine the nitric acid by Eder's method. This process is difficult, owing to irregularity in the combustion, and to the tendency of the tubes to break. Nonvolatile nitro-derivatives and ethereal nitrates may be converted into ammonia by the use of alkaline permanganate in an open dish, reduc-

tion of excess of the permanganate by alcohol, treatment of the diluted filtrate by ferrous sulphate, zinc dust and hydrochoric acid, and final distillation of the ammonia. Chem. Zeit., 14, 269,; Jour. Chem. Soc., 1891, 59, A, 109; Zeitschr. Anal. Chem., 1890, 458.

(45) WELLS, H. L. *Automatic Sprengel pump.*—The essentially new feature is a ram which forces the mercury into the reservoir by water pressure. Ber. Deut. Chem. Ges., 1891, 24, 1037–1040.

(46) ZALOZIECKI, R. *α Naphthol-Benzöin as an indicator.*—The writer describes the preparation of the reagent from α naphthol and benzo-trichloride in presence of benzol. It is colored an intense green by alkalies, and reddish-yellow by acids. It responds very promptly to carbonic acid, far more quickly than phenol-phthalein, which it otherwise closely resembles in its reactions. Chem. Zeit., 14, 605; Zeitchr. Anal. Chem., 1891, 30, 333–4; Journ. Anal. Chem., 4, 315; Vtjsschr. Chem. Nahr., 1890, 5, 231.

STUDY OF METHODS, 1890–'91.

In accordance with the instructions of last year, your reporter issued a circular asking the cooperation of a limited number of stations in a somewhat more detailed study of methods than we have usually made, instead of seeking a more extended cooperation covering fewer points.

The following extract from the circular of instruction shows the scope of the work contemplated:

I send herewith four samples: No. 1 is composed of nitrates; No. 2, of dissolved bone and cotton-seed meal; No. 3, of dissolved bone, cotton-seed meal, and nitrates; No. 4, of ground bone.

"In addition to the usual determinations of moisture and of nitrogen by the soda lime, absolute, Ruffle, and Kjeldahl methods, as recommended by the A. O. A. C., in 1890, I would suggest the following subjects as worthy of your study:

"(1) Comparison of methods of 1889–'90, as respects the use of 2 grs. of zinc sulphide and 1 gr. of salicylic acid, instead of 2 grs. of zinc dust and 2 grs. of salicylic acid, in Scovell's modification of the Kjeldahl method.

"(2) Comparison of the use of metallic mercury instead of mercuric oxide in the same.

"(3) Comparison of sodium hyposulphite and hypophosphite (3–5 gr. of the crystallized salts) as reducing agents in the place of zinc dust or zinc sulphide. (Refer to Foerster, Landw. Vers. Stat., 38, 165–194.)

"(4) The use of the Wilfarth modification, using 200 cc. of phosphoric acid per liter of the acid, for the Kjeldahl method.

"(5) The use of the Gunning method in the absence of nitrates. (See proceedings of this association for 1890, p. 18; also Zeitschr. Anal. Chem., 28, 188–191.)

"Accompanying the samples is a sealed bottle of our decinormal sulphuric acid, which please compare with your own standard acid used in this work."

Owing to pressure of other duties, to the difficulties encountered in a portion of the work, and to the number of points indicated, the data received do not cover the points indicated so fully as it was hoped they might; nevertheless, many points are well covered.

The reports received represent the work of the following analysts:

Arkansas: Mr. George L. Teller;

Connecticut, New Haven: Messrs. A. L. Winton and A. W. Ogden.

Kentucky: Messrs. Alfred M. Peter and H. E. Curtis;

Maryland: Mr. H. J. Patterson;

New Jersey, State Station: Mr. E. B. Voorhees;

Rutgers College Station: Mr. Charles S. Cathcart;

North Carolina: Messrs. B. W. Kilgore and F. B. Carpenter;

Pennsylvania: Mr. George L. Holter;

West Virginia: Messrs. Rudolf de Roode and B. H. Hill;

U. S. Department of Agriculture: Messrs. T. C. Trescot and A. E. Knorr.

STANDARD ACID.

Before proceeding to the discussion of the analytical data accumulated, there are certain points of interest to be noted touching the standard acid.

The standard acid sent out by your reporter was prepared so as to agree strictly with the standard hydrochloric acid of the Pennsylvania station. The standardization of this acid was performed by Mr. George L. Holter, who also performed, under the general supervision of the writer, all the analytical work upon nitrogen methods reported from the Pennsylvania station, and to whom the writer is indebted for many observations.

The results of standardization may be summarized as follows:

(1) *By silver nitrate.*—Carefully weighed portions of the $\frac{N}{10}$ HCl were precipitated by silver nitrate, washed completely, dried and weighed. These operations were all performed at night by gaslight. Assuming the specific gravity of the acid to be 1.000 at 15° C., the mean of these determinations showed the acid to be 99.87 per cent of normal strength, the range being only 0.03 per cent. Correcting for a specific gravity of 1.0015 at 15° C., the strength would be 100.02 per cent of normal.

(2) *By barium sulphate.*— A decinormal sulphuric acid solution was prepared strictly comparable with the hydrochloric acid by the readings of a carefully calibrated burette, reading to twentieths of a cubic centimeter. Portions of 25 cc. of this solution were weighed and precipitated as barium sulphate with the usual precautions. The mean result, uncorrected for specific gravity of the acid, showed the latter to be 99.97 per cent of normal, or, corrected for density, 100.30 per cent.

(3) *By electrolysis of copper sulphate (Hart and Croasdale's method).*—For this purpose recrystallized chemically pure copper sulphate, finely pulverized and dried at 110° C., was used; 5.1956 grs. of this sulphate yielded 1.3087 grs. of pure copper. The residual acid was diluted to 415.6 grs., of which 20 cc., assumed to weigh 20 grs., required for neutralization 19.8 cc. of decinormal alkali, corresponding exactly to the decinormal hydrochloric acid. The total weight of sulphuric acid set free was therefore 2.0177 grs. The copper found is 99.14 per cent of the calculated amount. The acid found is 98.84 per cent of the calculated amount. These figures indicate that the salt used was pure, and the slight discrepancy may be attributed either to slight oxidation of the copper, or to excess of the decinormal hydrochloric above normal strength. If to the latter, the excess would be only 0.3 per cent; but the former is more probably the cause. The fact that only 99 per cent of the calculated amount of copper was obtained may be due either to incomplete electrolysis, or less probably, to failure to drive off all the moisture in drying. Since, however, as the standardization is based upon the weight of the pure copper obtained, neither of these causes would affect the accuracy of the process, the original weight of the salt electrolyzed would be needed simply as a check upon the purity of the salt.

As a further test of the accuracy of this method, a number of determinations were made using undetermined quantities of the above sulphate for electrolysis, the weight of the copper and the volume of the solution of sulphuric acid were noted, and also the amount of alkali standardized by the decinormal hydrochloric acid required for neutralization of 20 cc. of the sulphuric acid. The results stated in per cent of the normal, as indicated by the hydrochloric acid, are as follows:

	Per cent.		Per cent.
1	99.62	4	99.70
2	100.13	5	99.66
3	99.91	6	99.66

With very careful manipulation the sulphuric acid obtained may fall 0.4 per cent below normal, or as a mean of six determinations 0.22 per cent. This is due probably to a very slight oxidation of the thin coat of copper on the platinum electrode during drying. The method therefore gives results safely within the limits of error

of the most careful determination of nitrogen by any of the methods in use, even where highly nitrogenous substances are subjected to analysis. It is simple, and undoubtedly affords an excellent check upon acid standardization by other methods, though apparently not capable of as great accuracy as the silver nitrate method.

(4) The method by tetra-oxalate of potash was not employed because it was found impossible to get a perfectly satisfactory salt, and because, in our experience, even with dimethyl-aniline orange as an indicator, it was found impossible to get a sharp end reaction. A tetra-oxalate prepared under my direction last year by Dr. McDonnell by dissolving together the molecular proportion of chemically pure acid potassium oxalate and oxalic acid, gave after the fourth crystallization results showing it to possess an acidity 1.17 per cent greater than that sent out by Dr. Caldwell for comparison of standards, and which both the latter and Mr. Scovell stated to agree exactly in strength with their acids standardized by silver nitrate.

Another sample obtained from Baker and Adamson, when precipitated as calcium oxalate and weighed as CaO, gave only 96.51 per cent of the calculated amount of the latter oxide as a mean of three closely concordant determinations. In all cases Clarke's revised table of atomic weights was taken as the basis of computation.

It will be of interest to compare the above results with those reported by other analysts, after comparison with their own acids, expressing all in terms of the normality of the acid sent out:

	Per cent.
(1) Holter: By silver nitrate	100.02
By barium sulphate	100.30
By electrolysis of copper sulphate	99.77
(2) Peter: (Pipette not calibrated)	99.49
(3) Teller: By silver nitrate	100.00
(4) Trescot:[1] By silver nitrate	100.00
(5) De Roode: By silver nitrate	100.00
(6) Jenkins: By silver nitrate and sodium carbonate	100.06
(7) Voorhees:[2] By silver nitrate	100.41
(8) Patterson found it slightly above normal but did not report exact figures.	
(9) Battle: By silver nitrate and ammonium chloride	100.00

PREPARATION OF SAMPLES.

The materials used were:

I. Commercial sodium nitrate.
II. C. P. potassium nitrate.
III. Dissolved bone, from a lot kindly furnished for the purpose by Dr. Terne.
IV. Cotton-seed meal.
V. Ground bone.

The mixtures were composed:

(1) Of 1 part of sodium nitrate to 2 parts of the potassium nitrate.
(2) Of 1 part cotton-seed meal to 2 parts dissolved bone.
(3) Of 1 part potassium nitrate, 1 part cotton-seed meal, and 3 parts dissolved bone.
(4) Of simple ground bone.

The nitrates were thoroughly pulverized before mixing, and the cotton-seed meal, and the ground and dissolved bones, were put through a sieve with 60 meshes to the inch, and each made thoroughly homogeneous before weighing; after combining, the several samples were mixed till thoroughly homogeneous, and immediately put away in air-tight jars.

The composition of the ingredients is shown by the following tables, which are also arranged so as to show separately the results obtained by the several methods employed.

[1] Within limits of error of readings taken.

[2] Somewhat in question, as accident prevented the closest comparison desirable.

In calculating these results, the decinormal acid was accepted as strictly accurate, and corrections were made for reagents used in all cases, except that no blank determinations were made with the sodium hyposulphite, hypophosphite, and sulphate employed. The elimination of errors introduced by reagents is essential to a fair comparison of methods, even though the degree of accuracy of the method itself may vary with the amount of the substance taken for analysis to such a degree as to cover up the errors thus introduced.

The methods used were strictly official, except where deviations are indicated, and with the exception that cochineal was used as an indicator; no difficulty being experienced in getting a satisfactory end reaction when the soda used in the Kjeldahl work was fairly free from carbonate.

I.—ANALYSES OF DISSOLVED BONE AND COTTON-SEED MEAL.

Method of analysis.	Dissolved bone.			Cotton-seed meal.		
	No. of determinations.	Range.	Mean.	No. of determinations.	Range.	Mean.
		Per ct.	*Per cent.*		*Per ct.*	*Per ct.*
(*a*) Kjeldahl (with mercury, and without $K_2Mn_2O_8$)	7	.08	2.84	6	.08	7.73
(*b*) Soda lime	1	.....	2.83	1	.....	7.76
(*c*) Absolute	.....	.....	.....	1	.....	7.92
(*d*) Gunning (with mercuric oxide and $K_2Mn_2O_8$)	3	.04	2.78	3	.16	7.70

II.—ANALYSES OF SODIUM AND POTASSIUM NITRATES.

Method of analysis.	Sodium nitrate (com'l).			Potassium nitrate (c.p).		
	No. of determinations.	Range.	Mean.	No. of determinations.	Range.	Mean.
		Per ct.	*Per cent.*		*Per ct.*	*Per ct.*
(*a*) Scovell (mercury and zinc sulphide, without $K_2Mn_2O_8$)	4	.17	16.36	3	.06	13.72
(*b*) ' oerster-Scovell (mercuric oxide, hyposulphite, with $K_2Mn_2O_8$)	3	.11	16.36	3	.16	13.76
(*c*) Burney-Scovell (mercuric oxide, hypophosphite, with $K_2Mn_2O_8$)	2	.05	16.39	3	.07	13.79
Calculated as chemically pure	.....	.....	16.47	.....	.....	13.84

It is of interest in this connection to note that without correction for error introduced by reagents, the results yielded in the cases of the nitrates by methods *a*, *b*, and *c*, respectively, were sodium nitrate, 16.49, 16.44, and 16.47; potassium nitrate, 13.85, 13.84, and 13.87. These results are in remarkable accord with those of Mr. Woods and Mr. Vorhees, to which your attention was called last year. Prof. Peter analyzed C. P. potassium nitrate by Foerster's modification of Scovell's method, using mercuric oxide, and obtained as a mean of four results, 13.65 per cent, with a range of .07 per cent. The amount of the correction I do not know.

Accepting the results by the Kjeldahl and Scovell methods as correct, the composition of mixtures 1, 2, and 3 would be: 1, 14.60 per cent; 2, 4.47 per cent; 3, 5.99 per cent. Giving the nitrates their theoretical composition, the calculated composition of 1 and 3 would be 14.72 per cent and 6.02 per cent, respectively.

ANALYSES OF MIXTURES.

The samples were, as far as possible, bottled at the same time and carefully sealed, so that, unless exposed for a long time, or under very exceptional conditions, no great

difference in moisture would be anticipated. Moisture determinations have not been reported in all cases.

In most instances the moistures were determined by drying four or five hours in a drying oven. The results obtained by different analysts by this method were highly comparable, except in the case of the ground bone, where three analysts get 4.27 per cent and three others 3.22 per cent. The close agreement in other samples renders it probable that this lack of agreement was not due to difference in exposure and humidity of the atmosphere of the different laboratories. Mr. Knorr made the determination in a current of hydrogen at "steam heat" for four hours, and got higher results, as follows:

	2.	3.	4.
	Per ct.	*Per ct.*	*Per ct.*
Mean for six analysts, drying in air ovens	3.73	2.78	*4.27
Knorr, drying in hydrogen at "steam heat"	6.61	5.11	5.36

*Accepting the three highest.

Owing to the difference in method used in this case, it has not seemed desirable to make any correction for these differences in moisture.

The percentages of nitrogen reported are presented in the following tables. Except where indicated, corrections have been made for difference in standard acid, and for error introduced by reagents; such omissions of correction are indicated by an asterisk, and a double asterisk for the standard acid and the reagents, respectively.

III.—ANALYSES OF MIXTURES 2 AND 4, FREE FROM NITRATES.

Analysts.	2.			4.		
	No. of determinations.	Range.	Mean.	No. of determinations.	Range.	Mean.
I. KJELDAHL METHOD.						
(*a*) WITH MERCURIC OXIDE AND PERMANGANATE.		*Per ct.*	*Per ct.*		*Per ct.*	*Per ct.*
G. L. Holter (Pennsylvania Station)	5	0.18	4.42	4	0.06	2.33
A. M. Peter (Kentucky Station)	2	0.02	4.40	2	0.06	2.34
H. E. Curtis (Kentucky Station)	2	0.02	4.42	2	0.03	2.31
G. L. Teller (Arkansas Station)	(?)	(?)	4.42	(?)	(?)	2.34
A. L. Winton (Connecticut Station, New Haven)	2	0.05	4.49	2	0.07	2.31
A. W. Ogden (Connecticut Station, New Haven)	2	0.11	4.48	2	0.05	2.36
B. W. Kilgore (North Carolina Station)	2	0.01	4.47	1	.....	2.38
F. B. Carpenter (North Carolina Station)	2	0	4.50	2	0.05	2.38
T. C. Trescot (U. S. Department of Agriculture)	(?)	(?)	4.57	(?)	(?)	2.38
Means	.....	.....	4.46	.....	.....	2.35
Number of analysts	.....	.....	9	.....	.....	9
Minimum number of analyses	.....	.....	19	.....	.....	17
Extreme range of analysts' averages (per cent)	.....	.....	0.17	.....	.....	0.07
(*b*) WITH MERCURIC OXIDE, WITHOUT PERMANGANATE.						
G. L. Holter (Pennsylvania Station)	8	0.12	4.42	.....	.....	.....
H. J. Patterson (Maryland Station)	(?)	(?)	4.35	.....	.....	.....
Mean	.....	.....	4.39	.....	.....	.....
Number of analysts	.....	.....	2	.....	.....	.....
Minimum number of analyses	.....	.....	9	.....	.....	.....
Extreme range of analysts' averages (per cent)	.....	.....	0.07	.....	.....	.....

III.—ANALYSES OF MIXTURES 2 AND 4, FREE FROM NITRATES—Continued.

Analysts.	2.			4.		
	No. of determinations.	Range.	Mean.	No. of determinations.	Range.	Mean.
I. KJELDAHL METHOD—Continued.						
(*c*) WITH METALLIC MERCURY AND PERMANGANATE.						
		Per ct.	*Per ct.*		*Per ct.*	*Per ct.*
G. L. Holter (Pennsylvania Station)	4	0.08	4.47	2	0.03	2.30
H. E. Curtis (Kentucky Station)	1	.....	4.40	1	.....	2.31
R. de Roode (West Virginia Station)	2	0.02	4.47	3	0.04	2.34
B. H. Hill (West Virginia Station)	2	0.01	4.47	2	0.01	2.35
Means	.....	.....	4.45	.....	.....	2.33
Number of analysts	.....	.....	4	.....	.....	4
Minimum number of analyses	.....	.....	9	.....	.....	8
Extreme range of analysts' averages (per cent)	.....	.....	0.07	.....	.....	0.05
(*d*) WITH METALLIC MERCURY, WITHOUT PERMANGANATE.						
G. L. Holter (Pennsylvania Station)	.....	.....	.....	4	0.03	2.30
H. J. Patterson (Maryland Station)	(?)	(?)	4.49	(?)	(?)	2.52
E. B. Voorhees (New Jersey State Station)	3	0.09	4.39	4	0.05	2.45
C. S. Cathcart (Rutgers College Station)	(?)	(?)	4.49	(?)	(?)	2.44
Means	.....	.....	4.46	.....	.....	2.43
Number of analysts	.....	.....	3	.....	.....	4
Minimum number of analyses	.....	.....	5	.....	.....	10
Extreme range of analysts' averages (per cent)	.....	.....	0.10	.....	.....	0.22
II. SODA-LIME METHOD.						
G. L. Holter (Pennsylvania Station)	6	0.14	4.52	4	0.09	2.37
T. C. Trescot (U. S. Department of Agriculture)	(?)	(?)	4.43	(?)	(?)	2.31
Means	.....	.....	4.48	.....	.....	2.34
Number of analysts	.....	.....	2	.....	.....	2
Minimum number of analyses	.....	.....	7	.....	.....	5
Extreme range of analysts' averages, (per cent)	.....	.....	0.09	.....	.....	0.06
III. RUFFLE METHOD.						
T. C. Trescot (U. S. Department of Agriculture)	(?)	(?)	4.47	(?)	(?)	2.39
B. W. Kilgore (North Carolina Station)	1	.....	4.49	1	.....	2.34
F. B. Carpenter (North Carolina Station)	1	.....	4.50	1	.....	2.36
Means	.....	.....	4.49	.....	.....	2.36
Number of analysts	.....	.....	3	.....	.....	3
Minimum number of analyses	.....	.....	3	.....	.....	3
Extreme range of analysts' averages (per cent)	.....	.....	0.03	.....	.....	0.05
IV. ABSOLUTE METHOD.						
G. L. Holter (Pennsylvania Station)	2	0.09	4.52	2	0.07	2.35
T. C. Trescot (U. S. Department of Agriculture)	(?)	(?)	4.40	(?)	(?)	2.28
Means	.....	.....	4.46	.....	.....	2.32
Number of analysts	.....	.....	2	.....	.....	2
Minimum number of analyses	.....	.....	3	.....	.....	3
Extreme range of analysts' averages (per cent)	.....	.....	0.12	.....	.....	0

III.—ANALYSES OF MIXTURES 2 AND 4, FREE FROM NITRATES—Continued.

Analysts.	2.			4.		
	No. of determinations.	Range.	Mean.	No. of determinations.	Range.	Mean.
V. GUNNING'S METHOD.						
(*a*) PLAIN.						
		Per ct.	*Per ct.*		*Per ct.*	*Per ct.*
H. J. Patterson (Maryland Station)	(?)	(?)	4.38	(?)	(?)	2.25
R. de Roode (West Virginia Station)	3	0.02	4.47	2	0.01	2.33
Means			4.43			2.29
Number of analysts			2			2
Minimum number of analyses			4			3
Extreme range of analysts' averages (per cent)			0.09			0.12
(*b*) WITH MERCURIC OXIDE AND PERMANGANATE.						
G. L. Holter (Pennsylvania Station)	4	0.11	4.42	3	0.05	2.30
Number of analysts			1			1
Number of analyses			4			3

IV.—ANALYSES OF MIXTURES 1 AND 3, CONTAINING NITRATES.

Analyst.	1.			3.		
	No of determinations.	Range.	Mean.	No. of determinations.	Range.	Mean.
I. SCOVELL-KJELDAHL METHOD.						
(*a*) MERCURIC OXIDE, ZINC DUST (2 GRS.), SALICYLIC ACID (2 GRS.).						
(*aa*) *With permanganate.*						
		Per ct.	*Per ct.*		*Per ct.*	*Per ct.*
G. L. Holter (Pennsylvania Station)	5	0.05	14.59			
A. M. Peter (Kentucky Station)	5	0.09	14.35	2	0.02	5.94
H. E. Curtis (Kentucky Station)	2	0.02	14.31	2	0.06	5.91
A. L. Winton (Connecticut Station; New Haven)	2	0.03	14.52	2	0.04	5.91
A. W. Ogden (Connecticut Station; New Haven)	1		14.48	2	0.06	5.89
B. W. Kilgore (North Carolina Station)	3	0.12	14.69	2	0.02	6.07
F. B. Carpenter (North Carolina Station)	3	0.09	14.69	2	0.02	6.08
Mean			14.52			5.97
Mean of six			14.58			
Number of analysts			7			6
Minimum number of analyses			2			12
Extreme range of analysts' averages (per cent)			0.38			0.19
(*ab*) *Without permanganate.*						
G. L. Holter (Pennsylvania Station)	3	0.04	14.62	8	0.14	5.95
Number of analysts			1			1
Minimum number of analyses			3			8
(*b*) METALLIC MERCURY, ZINC DUST (2 GR.), AND SALICYLIC ACID (2 GR.)						
(*ba*) *With permanganate.*						
H. E. Curtis (Kentucky Station)	1		14.27	1		5.97
Number of analysts			1			11
Minimum number of analyses			1			

IV.—ANALYSES OF MIXTURES 1 AND 3, CONTAINING NITRATES—Continued.

Analyst.	1.			3.		
	No. of determinations.	Range.	Mean.	No. of determinations.	Range.	Mean.
I. SCOVELL-KJELDAHL METHOD—Continued.						
(*b*) METALLIC MERCURY, ZINC DUST (2 GR.) AND SALICYLIC ACID (2 GR.)—continued.						
(*bb*) *Without permanganate.*						
		Per ct.	*Per ct.*		*Per ct.*	*Per ct.*
H. J. Patterson (Maryland Station)	(?)	(?)	14.62	(?)	(?)	5.96
E. B. Voorhees (New Jersey State Station)	4	0.14	14.40	2	0.00	5.92
C. S. Cathcart (Rutgers College Station)	(?)	(?)	14.42	(?)	(?)	5.97
Mean			14.48			5.95
Number of analysts			3			3
Minimum number of analyses			6			4
Extreme range of analysts' averages (per cent)			0.22			0.05
(*c*) MERCURIC OXIDE, ZINC SULPHIDE (2 GR.) AND SALICYLIC ACID (1 GR.).						
(*ca*) *With permanganate.*						
G. L. Holter (Pennsylvania Station)				3	0.02	5.91
A. M. Peter (Kentucky Station)	2	0.01	14.47	3	0.11	5.97
G. L. Teller (Arkansas Station)	(?)	(?)	14.19	(?)	(?)	5.81
T. C. Trescot (U. S. Department of Agriculture)	(?)	(?)	14.51	(?)	(?)	5.84
Mean			14.39			5.88
Number of analysts			3			4
Minimum number of analyses			4			8
Extreme range of analysts' averages (per cent)			0.32			0.16
(*cb*) *Without permanganate.*						
G. L. Holter (Pennsylvania Station)				3	0.05	5.92
(*d*) METALLIC MERCURY, ZINC SULPHIDE, (2 GR.) AND SALICYLIC ACID (1 GR.).						
(*da*) *With permanganate.*						
G. L. Holter (Pennsylvania Station)	3	0.17	14.32			
R. de Roode (West Virginia Station)	3	0.06	14.59	3	0.10	5.96
B. H. Hill (West Virginia Station)	2	0.01	14.54	2	0.00	5.99
Mean			14.48			5.92
Number of analysts			3			2
Minimum number of analyses			8			5
Extreme range of analysts' averages (per cent)			0.27			0.03
(*db*) *Without permanganate.*						
G. L. Holter (Pennsylvania Station)	3	0.08	14.59	2	0.05	5.79
H. J. Patterson (Maryland Station)				(?)	(?)	5.73
E. B. Voorhees (New Jersey State Station)	5	0.18	14.29	2	0.07	5.83
C. S. Cathcart (Rutgers College Station)	(?)	(?)	14.26	(?)	(?)	5.95
Mean			14.38			5.81
Number of analysts			3			4
Minimum number of analyses			9			6
Extreme range of analysts' averages (per cent)			0.33			0.20

IV.—ANALYSES OF MIXTURES 1 AND 3, CONTAINING NITRATES—Continued.

Analyst.	1.			3.		
	No. of determinations.	Range.	Mean.	No. of determinations.	Range.	Mean.
II. FOERSTER-SCOVELL-KJELDAHL METHOD.						
(*a*) MERCURIC OXIDE, PERMANGANATE, AND SODIUM HYPOSULPHITE (5 GR.).						
(*aa*) *With salicylic acid* (2 *gr.*).		*Per ct.*	*Per ct.*		*Per ct.*	*Per ct.*
G. L. Holter (Pennsylvania Station)	4	0.16	14.48	4	0.09	5.82
A. M. Peter (Kentucky Station)	2	0.02	14.53	3	0.07	6.00
H. E. Curtis (Kentucky Station)	3	0.06	14.47	2	0.00	5.90
Mean			14.49			5.91
Number of analysts			3			3
Minimum number of analyses			9			9
Extreme range of analysts' averages (per cent)			.06			0.18
(*ab*) *With salicylic acid* (1 *gr.*).						
A. M. Peter (Kentucky Station)	2	0.00	14.50			
III. BURNEY-SCOVELL-KJELDAHL METHOD.						
MERCURIC OXIDE, SALICYLIC ACID (2 GR.), PERMANGANATE, AND SODIUM HYPOPHOSPHITE (5 GR.).						
G. L. Holter (Pennsylvania Station)	3	0.09	14.52	3	0.16	5.82
IV. RUFFLE'S METHOD.						
G. L. Holter (Pennsylvania Station)	2	0.05	14.65	2	0.02	5.95
T. C. Trescot (U. S. Department of Agriculture)	(?)	(?)	14.58	(?)	(?)	5.96
B. W. Kilgore (North Carolina Station)				1		5.96
F. B. Carpenter (North Carolina Station)				1		6.04
Mean			14.62			5.98
Number of analysts			2			4
Minimum number of analyses			3			5
Extreme range of analysts' averages (per cent)			0.07			0.08
V. ABSOLUTE METHOD.						
G. L. Holter (Pennsylvania Station)				2	0.09	.17
T. C. Trescot (U. S. Department of Agriculture)	(?)	(?)	14.68	(?)	(?)	5.98
Mean			14.68			6.08
Number of analysts						2
Minimum number of analyses						3
Extreme range of analysts' averages (per cent)						0.19
VI. ULSCH'S METHOD.						
T. C. Trescot (U. S. Department of Agriculture)	(?)	(?)	14.61			
H. J. Patterson (Maryland Station)	(?)	(?)	14.51			
Mean			14.56			
Number of analysts			2			
Minimum number of analyses			2			
Extreme range of analysts' averages (per cent)			0.10			
VII. KJELDAHL, WITHOUT REDUCING AGENTS.						
(*a*) MERCURIC OXIDE WITHOUT PERMANGANATE.						
H. J. Patterson (Maryland Station)				(?)	(?)	4.00
(*b*) METALLIC MERCURY, WITHOUT PERMANGANATE.						
H. J. Patterson (Maryland Station)				(?)	(?)	4.08
VIII. GUNNING'S METHOD.						
H. J. Patterson (Maryland Station)				(?)	(?)	2.90

In order to show at a glance the salient features of the preceding tables, the following summary has been prepared:

V.—SUMMARY OF RESULTS ON SAMPLES NOT CONTAINING NITRATES.

Methods.	2.				4.			
	Means.	Number of analysts.	Minimum number of analyses.	Extreme range of analysts' averages.	Means.	Number of analysts.	Minimum number of analyses.	Extreme range of analysts' averages.
	Per ct.			*Per ct.*	*Per ct.*			*Per ct.*
Calculated composition	4.17	.....	.....	.....	.....	.....	.....	.....
I. Kjeldahl method:								
(*a*) With mercuric oxide and permanganate	4.46	9	19	0.17	2.35	9	17	0.07
(*b*) With mercuric oxide; without permanganate	4.39	2	9	0.07	.....	.....	.....	.....
(*c*) With metallic mercury and permanganate	4.45	4	9	0.07	2.33	4	8	0.05
(*d*) With metallic mercury without permanganate	4.46	3	5	0.10	2.43	4	10	0.22
Mean for Kjeldahl method in general	4.45	14	42	0.22	2.36	13	35	0.22
II. Soda-lime method	4.48	2	7	0.09	2.34	2	5	0.06
III. Ruffle method	4.49	3	3	0.03	2.36	3	3	0.05
IV. Absolute method	4.46	2	3	0.12	2.32	2	3	0.07
V. Gunning's method:								
(*a*) Plain	4.43	2	4	0.09	2.29	2	3	0.12
(*b*) With addition of mercuric oxide and permanganate	4.42	1	4	.....	2.30	1	3	.....

VI.—SUMMARY OF RESULTS ON SAMPLES CONTAINING NITRATES.

Methods.	1.				3.			
	Means.	Number of analysts.	Minimum number of analyses.	Extreme range of analysts' averages.	Means.	Number of analysts.	Minimum number of analyses.	Extreme range of analysts' averages.
	Per ct.			*Per ct.*	*Per ct.*			*Per ct.*
Calculated composition	[1]14.00	.....	.....	.....	[1]5.99	.....	.....	.....
I. Scovell-Kjeldahl method:								
(*a*) Mercuric oxide, zinc dust, and salicylic acid:								
(*aa*) With permanganate	[2]14.52	7	21	0.38	5.97	6	12	0.19
(*ab*) Without permanganate	14.62	1	3	.....	5.95	1	8	.....
(*b*) Metallic mercury, zinc dust, and salicylic acid:								
(*ba*) With permanganate	14.27	1	1	.....	5.97	1	1	.....
(*bb*) Without permanganate	14.48	3	6	0.22	5.95	3	4	0.05

[1] Holter's results by Scovell method—mercury and zinc sulphide without permanganate.

[2] Mean for six analysts, 14.58 per cent, with a range of 0.28 per cent.

VI.—SUMMARY OF RESULTS ON SAMPLES CONTAINING NITRATES—Continued.

Methods.	1.				3.			
	Means.	Number of analysts.	Minimum number of analyses.	Extreme range of analysts' averages.	Means	Number of analysts.	Minimum number of analysts.	Extreme range of analysts' averages.
I. Scovell-Kjeldahl method—Continued.								
(*c*) Mercuric oxide, zinc sulphide, and salicylic acid:	*Per ct.*			*Per ct.*	*Per ct.*			*Per ct.*
(*ca*) With permanganate	14.39	3	4	0.32	5.88	4	8	0.16
(*cb*) Without permanganate	.....	.....	.....	.....	5.92	1		.....
(*d*) Metallic mercury, zinc sulphide, and salicylic acid:								
(*da*) With permanganate	14.48	3	8	0.27	5.92			0.03
(*db*) Without permanganate	14.38	3	9	0.33	5.81		6	0.20
Mean for Scovell-Kjeldahl method in general	14.46	14	52	0.50	5.96	14	47	0.35
II. Foerster-Scovell-Kjeldahl method: Salicylic acid, mercuric oxide, permanganate, and sodium hyposulphite:								
(*a*) With 1 gr. of salicylic acid	14.49	3	9	0.06	5.91	3	9	0.18
(*b*) With 2 grs. of salicylic acid	14.50	1	2	.....	.....	.....	.....	.....
Mean for Foerster's method.	14.49	3	11	0.06	5.91	3	9	0.18
III. Burney-Scovell-Kjeldahl method: Mercuric oxide, salicylic acid, permanganate, and sodium hypophosphite	14.52	1	3	.....	5.82	1	3	.....
IV. Ruffle method	14.62	2	3	.07	5.98	4	5	0.03
V. Absolute method	14.68	1	1	.....	6.08	2	3	0.19
VI. Ulsch's method	14.56	2	2	.10	.....	.....	.....	.....
VII. Plain Kjeldahl:								
(*a*) Mercuric oxide	.....	.....	.....	.....	4.00	1	1	.....
(*b*) Metallic mercury	.....	.....	.....	.....	4.08	1	1	.....
VIII. Plain Gunning	.....	.....	.....	.....	2.90	1	1	.....

So far as the agreement between different analysts is concerned, the excellent agreement of the standard acids is a matter for congratulation; and where the same methods were pursued the differences in results fall much below those reported last year. To take the widest variation, for example, in sample No. 1, pure nitrate, the modified Kjeldahl method, without reference to differences of detail, showed a maximum range between analysts' averages of 0.50 per cent this year, as compared with a range of 0.80 per cent last year, the number of analysts represented being 22 and 14,

respectively; but this year on the same fertilizer, where exactly the same details were adopted, the extreme range between averages is 0.38 per cent. With mixed organic and nitric nitrogen the extreme range last year by this general method was 1.04 from 21 chemists, and this year 0.35 from 14 chemists. With the Kjeldahl method on non-nitrated goods last year the extreme range was 0.70 per cent from 22 chemists; this year, without reference to detail, 0.22 from 14 analysts; or, taking details into account, 0.17 from 9 analysts. The mean range by the use of the Kjeldahl plain and modified, soda lime, and Ruffle methods, on 3 samples was 0.62 per cent; this year, by the same methods, on 4 samples, the mean of the extreme ranges is 0.167 per cent, where the differences in detail of the several Kjeldahl methods are not taken into consideration. In spite of this improvement the results show the need in a number of cases of a more careful perfection of methods by the study of details upon a material of definite composition.

Although the results have been classified with the purpose of bringing out more clearly the effects of the use of the several details recommended in the various official methods, the accuracy of comparison is impaired by the introduction of different personal equations, since the different details were not all studied by each chemist, but one by some, others by others. This fact must be clearly recalled in drawing conclusions from the averages presented, and will be offset, as far as the material will allow, by comparing the work of the same analysts on the several details.

First, as to the several details of the Kjeldahl and modified Kjeldahl methods: The use of permanganate does not seem to have been of any benefit in the case of plain goods. Mr. Holter obtained identical results with or without it, and the difference in combined results is slight and variable. The same is true in case of the nitrated goods, although the variations are wider: but this is, in some instances, due to the smaller number of analysts reporting. The results are certainly sufficiently numerous and concordant to disprove the necessity for its use in the case of the various goods made up of the ingredients used in the above samples. In the face, however, of strong statements in opposition to this conclusion, your reporter does not feel that it would be wise to change the official method at this point until a wider range of substances has been carefully studied. Nevertheless, it should be noted that its use has already been discontinued by many of our chemists.

Metallic mercury *vs.* mercuric oxide: Here, too, there seems, so far as results go, to be little choice between the two substances. No disadvantage was noted from the use of metallic mercury in the case of food and fertilizer analyses, though one observer considers the handling of the oxide as more convenient: but its use in milk analysis, Mr. Holter remarks, is attended with great frothing, making its employment in that case objectionable.

Zinc sulphide *vs.* zinc dust as a reducing agent: The explosive violence with which the dust is attacked by the acid, with the consequent danger of loss by spurting, and by volatilization of nitrogen due to the rapid rise of temperature, necessitating its addition in small quantities, led to the suggestion of the use of zinc sulphide in its stead, since the latter was attended by neither of the above disadvantages. The results of individual chemists, as well as the average, seem to indicate that the sulphide does not accomplish the ammonification so completely as the zinc dust, except in a few instances. The difference is, with a single exception, slight in the case of organic goods containing over 1 per cent of nitrate nitrogen, but becomes quite considerable in case of most of the pure nitrate averages in which this difference of reducing agent occurs. On the other hand, Mr. Voorhees states that as a result of 66 analyses of nitrated organic goods in which the two agents were employed, the following averages were obtained: With zinc dust, 2.825 per cent; with zinc sulphide, 2.817 per cent. Both sets of results indicate that the use of the sulphide may be regarded as entirely safe in the case of goods such as No. 3, but as of doubtful desirability in case of pure nitrates. Fuller investigation is needed with reference to the latter class of goods before any certain conclusions are attainable, especially to determine the influence of a larger amount of salicylic acid on the results.

The substitution of 5 gr. of crystallized sodium hyposulphite for zinc dust, according to Foerster's suggestion, gave results fully as good as those obtained with the latter reagent, and the whole charge can be introduced at once, just as in the case of zinc sulphide, with even less heating than in the latter case, and without the formation of a sulphate readily solidifying out from the menstruum in the digestion flask. This should necessitate less attention and less washing where a transfer is made to another flask for distillation. Mr. Holter states that he has observed no peculiar mechanical difficulties in the use of this modification. Your reporter, therefore, suggests that as it gives good results both with pure nitrates and with mixed organic and nitric nitrogen, it may be safer than zinc sulphide in analyzing the former class of goods. Mr. Peters's results, though limited in range, seem to indicate that 1 gr. of salicylic acid may prove sufficient, as with zinc sulphide. The adoption of this method seems entirely safe.

The use of sodium hypophosphite, instead of zinc dust, is not so promising; for although its very limited trial gives fair results, Mr. Holter notes that there is so much frothing during digestion as to require the greatest care to prevent loss. Gunning's method gave results slightly lower than most others, but still very satisfactory. The addition of oxidizing agents did not increase the yield of nitrogen, and Mr. Holter observed that where they were used the material was often violently projected from the flask.

In general, the results on this class of methods indicate their very satisfactory applicability to the analysis, not only of purely organic or ammonium compounds, but also, under proper modifications, of those mixed with a considerable proportion of nitrates; but since in the case of pure nitrates the average result is so far below the calculated one, checked as the latter is by various determinations, there seems to be much wisdom in the action of the German chemists in recommending the use of other methods where such goods are to be analyzed, and while not condemning the method in the hands of many analysts, your reporter would suggest that in such cases the use of some other method would usually be preferable.

Among other points worthy of investigation there may be mentioned the possible formation of insoluble ammonio-zinc compounds where the zinc dust is used in distillation; and if that is not found to be the case, the possibility of dispensing with the addition of potassium sulphide for the decomposition of the mercurammonium compounds, relying upon the zinc dust for that purpose.

The results obtained by use of the soda-lime and Ruffle methods this year were very satisfactory and need no comment, unless it be that the attainment of satisfactory results in the case of No. 1 required a large dilution of the substance with sugar.

Ulsch's method, by reduction with reduced iron, makes an excellent showing in the few tests reported, and is said by the analysts testing it, to be very easy to perform, and to have given fully as excellent results when applied to other samples as those indicated in this report. Its thorough trial in a large number of laboratories during the coming year is suggested; for, in addition to simplicity, cheapness, quickness, and effectiveness, it has the merit of requiring little more than the apparatus already in use for the Kjeldahl method.

Your reporter recommends the following changes in the official methods:

On page 192, after the words "zinc sulphide," in the sixth line, add "or 5 gr. of crystallized sodium hyposulphite."

Also, that Gunning's method be admitted to the list of official methods in the case of non-nitrated goods.

The following suggestions are repeated:

(1) That a fuller comparison of zinc dust and zinc sulphide be made, especially with pure nitrates, so as to show more clearly whether the diminution in the quantity of aromatic acid has been wise where such large quantities of nitrogen are to be ammonified.

(2) The question as to the formation of insoluble ammonio zinc compounds when zinc dust is used in distillation, should be investigated.

(3) Likewise, the power of zinc dust to completely decompose mercurammonium compounds under the conditions obtaining in the Kjeldahl distillation, thus rendering it possible to omit the use of the potassium sulphide.

(4) That Ulsch's method receive a thorough trial with reference to its use as an official method for pure nitrates.

(5) That Hart and Croasdale's method of standardization be likewise thoroughly tested.

The experience of this year fully confirms the suggestion made last year, that although the check upon the work of individuals that is secured by the widely extended analyses of a few samples by a few methods is valuable, there is also much need for a more careful study of details, all of which should be tested by a considerable number of analysts, so as to permit the elimination of the personal equation, which is not possible when the different details have not been studied by the same individuals, as was too frequently the case this year. It would seem better, for the most part, to choose in any one laboratory for the cooperative work of a single year only a limited field, and investigate it thoroughly, while the first plan adopted by this association might very profitably be introduced every three or four years, to serve as a general check for analysts all over the country.

In conclusion, your reporter desires to express his appreciation of the prompt and interested attention which all the analysts coöperating in this work have shown.

The PRESIDENT. Are there any other papers on this subject?

Mr. VAN SLYKE. I have no paper, but am desirous of mentioning some results of a comparison between the Gunning modification of the Kjeldahl method and the regular Kjeldahl method, especially in connection with the analysis of fertilizers. The examined fertilizers were those occurring in ordinary work, and represented a large variety of the fertilizers sold in New York State. I found an average difference of 0.06 per cent. In only one case was the difference over 0.15 per cent; in this case it was 0.21 per cent. In only five or six cases did the difference exceed 0.10 per cent, while in over 13 per cent of the whole there was no difference whatever. I am partial to the Gunning method, and would like to see it adopted by the association.

Mr. Patrick presented the following paper:

A NEW DISTILLING FLASK FOR USE IN THE KJELDAHL PROCESS.

By G. E. PATRICK.

The only serious drawback to the Kjeldahl method of nitrogen determination is the breakage of distillation flasks, and in laboratories where many determinations of albuminoid nitrogen are made by the Stutzer process, this breakage is often a matter of much annoyance and considerable expense, since only the best quality of flasks will long stand the requirements of the process.

Some months ago the breakage in a certain lot of flasks purchased for this laboratory having become unendurable, it occurred to the writer to try copper flasks; and upon trial, the results have been so satisfactory that I can with confidence recommend the plan to other chemists.

The copper flasks used are the ordinary one-pint oxygen retorts, minus the caps, delivery tubes, and clamps.

Omitting here all record of test analyses, which will appear in a note shortly to be published in the Journal of Analytical Chemistry, suffice it to say, that the results are practically identical with those obtained by distilling from glass.

One or two details should be specially mentioned. I am in the habit of adding 30 cc. of K_2S solution instead of 25 cc., as laid down in the association method. Whether this increase is really necessary I can not say. I commenced making it, in my first

experiments, from fear that the copper of the flasks would rob the HgO of part of the sulphur intended for it, and have continued the custom without really proving its necessity. But as 25 cc. are often barely enough of the K_2S, even with glass flasks, the increase certainly seems reasonable.

The corrosion of the flasks by the sulphide will doubtless in time destroy them, but before that time arrives they will have paid for themselves many times over in saving of glassware and relief from anxiety.

The flasks are heated by a rather small naked flame; a large flame under the one-pint flask will boil the charge over.

The receiving flasks are marked at the 200 cc. level, to show when the operation is finished. No zinc or pumice is required to prevent bumping; otherwise the arrangements are as usual. The distillation is completed within thirty minutes; so that the saving of time is very great. (Experiment Station, Ames, Iowa, August 10, 1891.)

The PRESIDENT. The recommendations of the reporter will be taken up in the order presented.

Mr. Frear read the first recommendation: On page 193, sixth line. After the words, "then add direct 2 grs. of zinc sulphide," insert "or 5 grs. of crystallized sodium hyposulphite." Adopted.

The next recommendation, the addition of Gunning's method as an alternate method, was read.

Mr. SCOVELL. I think before this method is adopted there had better be some further experiments made with special reference to testing its accuracy. I have not found it altogether satisfactory.

Mr. VAN SLYKE. I have tested the method, and found it very satisfactory, and being satisfied that it is a reliable method, I am desirous of seeing it adopted as an alternate method.

Mr. FREAR. I have made the recommendation because I have heard but few complaints against the method, and have obtained nothing but good results in my own work, but nothing is to be lost by conservatism, and I would not object to having it tried for another year before adoption.

Mr. PAYNE. I move that the Gunning method be referred back to the reporter for trial another year. Carried.

Mr. PAYNE. I notice that on page 193, in the Ruffle method, mention is made of finely powdered crystallized sodium hyposulphite. I think most of the members, in using the Ruffle method, have gotten rid of the water of crystallization before beginning their work. The method makes no mention of that, and I think it would be confusing to a beginner. I move that the word "crystallized" be struck out, and after the words "sodium hyposulphite," the words "dried at 100° C.," be added. Carried.

Mr. VAN SLYKE. Is it desirable to retain dimethy-laniline orange as an indicator in standardizing the acid? It has not proven satisfactory in my laboratory.

Messrs. MCDONNELL and PAYNE. We have given it a trial, but did not find it satisfactory, and did not continue its use.

Mr. VAN SLYKE. I move that it be struck out. Carried.

Mr. DE ROODE. I think the method of standardizing by potassium

tetra-oxalate ought to be struck out. I have made inquiries concerning it among the members present, and a good many seem to think it very hard to prepare it in an absolutely pure state. I have prepared some carefully, but it still has too great an acidity. I ask for an expression of opinion in regard to this matter.

Mr. PAYNE. I have tried it and given it up. It is not satisfactory.

Mr. KILGORE. I have tried it, and found trouble in getting the end reaction.

Mr. ROSS. I have found it unsatisfactory when used in connection with ammonia, but with sodium hydrate solution and with phenolphthalein as an indicator it has been entirely satisfactory. The solutions used on the nitrogen determinations the previous year were worked up by solutions standardized by potassium tetra-oxalate, and the results were as satisfactory as could be desired.

Mr. DE ROODE. I have the same experience on that point, but could not get the tetra-oxalate pure.

The PRESIDENT. I have found no difficulty in making the tetra-oxalate pure, and have made a trial that seems satisfactory. It was put in the hands of 30 or 40 students working at analyses, and they were required to make the standardization by silver and by tetra-oxalate, using litmus as an indicator. The results agreed very closely. I have not tried it with ammonia. I want to say a word while speaking about the electrolosis of copper sulphate. I have found in testing students' work that they could not get good results when they took copper sulphate that has been air-dried. Anhydrous copper sulphate was then tried, and very close results were obtained, but it was difficult to get them with sulphate dried most carefully, powdered, and pressed between filter paper. I simply mention this as a bit of experience which may be of some value. In regard to the tetra-oxalate, as it could not be obtained pure, it might be better to leave it out.

Mr. PAYNE. I think it would be well to give up the tetra-oxalate, because the method prescribed the use of ammonia, and all who had ever used it with ammonia agree that it does not work well. I move that it be struck out. Carried.[1]

Mr. Battle presented the following report:

REPORT ON POTASH.

By H. B. BATTLE.

I am not able at this time to present any recent literature of value bearing on the determination of potash. Mr. A. E. Knorr, of the U. S. Department of Agriculture, has kindly consented to assist me in this respect, and if any be found it will be printed in the proceedings.[2]

[1] This motion was reconsidered, p. 153, and the method of standardization by tetra-oxalate continued for another year.—H. W. W.

[2] After a thorough search of the journal literature on this subject no references could be found bearing on points of interest so far as the work of this association is concerned.

In May, 1891, letters were written to twenty-one stations and official chemists, charged with fertilizer control, asking if they desired to cooperate in the examination of samples for potash determination. Fifteen of these replied in the affirmative, and to these three samples each were sent. Replies have only been received from ten official chemists, covering the work of eleven analysts. The three samples were as follows:

No. 1, ammoniated fertilizer, potash derived from tobacco stems, muriate and sulphate of potash.

No. 2, ammoniated fertilizer, potash derived from crude potash salts, cotton-seed meal and hull ashes, and muriate of potash.

No. 3, commercial sulphate of potash.

In the examination it was suggested that comparison be made with:

(*a*) The Lindo-Gladding method.

(*b*) The Alternate method.

(*c*) Treatment with H_2SO_4, and ignition for samples Nos. 1 and 2.

(*d*) The alternate method, making only one filtration. (See discussion Bulletin 28, p. 75, also resolution that the reporter request this comparison.)

(*e*) Any other work at the option of the cooperators

No determination of moisture was considered necessary. The samples were carefully ground to pass a sieve with 40 meshes to the linear inch, and most accurately mixed. The preparation was very carefully done, and the mixing is vouched for. Bottles were filled by dipping up portions from the carefully mixed mass. Analysts were requested to report such modifications in the present methods as seemed desirable, as well as all such matters as they desired to have the Association consider.

Fertilizers were chosen, containing cotton-seed meal and tobacco stems, as well as potash salts and cotton-seed hull ashes, for furnishing the potash material. As bearing upon the presence of potash in organic material, such as tobacco stems, I would like to refer to some experiments made under my direction by my assistant, Mr. J. R. Harris, in which a comparison is made with the total potash present, as determined by the previous ignition with H_2SO_4 and the water soluble potash. Tobacco from various States, Missouri, Kentucky, Ohio, Virginia, and North Carolina, was used. The total potash varied from 9.08 per cent down to 3.66 per cent. It is very noteworthy that over 90 per cent of this total potash in each sample was soluble in water. In one case as high as 98.09 per cent was noticed. Following is a record of these analyses, together with other determinations, inserted as a matter of interest:

	Moisture.	Volatile and organic matter.	Crude ash.	Phosphoric acid.	Ammonia.	Water-soluble potash.	Total potash.	Water-soluble in total potash.
	Per ct.	*Per ct.*	*Per ct.*	*Pr. ct.*	*Pr. ct.*	*Pr. ct.*	*Pr. ct.*	*Pr. ct.*
Missouri White Burly tobacco	16.56	61.65	21.79	.82	3.06	8.44	9.08	92.95
Kentucky White Burly tobacco	16.85	61.69	21.46	.72	2.89	8.36	8.89	94.04
Ohio White Burly tobacco	15.70	62.17	22.13	.84	3.47	8.11	8.56	94.63
Virginia stems for shipping or export tobacco	16.20	63.72	20.08	.56	3.14	6.35	7.00	90.71
Virginia shipping or export tobacco	17.15	63.80	19.05	.43	2.15	5.25	5.63	93.25
North Carolina Bright Legs cigarettes	18.18	65.86	15.96	.51	.93	4.97	5.33	93.15
North Carolina Bright Legs cigarettes short	15.90	65.80	18.30	.58	1.71	4.11	4.19	98.09
Stalk of North Carolina bright tobacco	17.36	71.78	10.86	52	2.11	3.92	4.34	90.32
North Carolina chewing tobacco	16.75	67.40	15.85	.73	1.19	3.37	3.66	92.08
North Carolina stems, short, from smoking tobacco	16.00	67.16	16.84	.59	1.14	4.80	5.14	93.38

The result of the examination of the three samples sent out is as follows:

SAMPLE 1.—TOBACCO STEMS, MURIATE AND SULPHATE OF POTASH.

Analysts.	Lindo-Gladding with H_2SO_4.		Lindo-Gladding, water solution.	Alternate, two filtrations.	Alternate, one filtration.
	Per cent.		*Per cent.*	*Per cent.*	*Per cent.*
J. S. Meng, Experiment Station, Mississippi	1.97 2.00	1.99	.....	.....	.....
W. S. Sweetser, Experiment Station, Pennsylvania.	[1]1.91 [2]2.03	1.90	.....	.....	.....
R. B. Moore, Experiment Station, Massachusetts...		1.74	.....	.....	.....
E. H. Farrington, Experiment Station, Illinois.....		2.20	.....	2.22	.....
A. L. Winton and O. W. Ogden, Experiment Station, Connecticut	1.64 1.65	1.65	[3]1.78 1.76 1.74 } [3]1.75	.....	.....
F. W. Morse, Experiment Station, New Hampshire.		1.66	.....	1.67	1.73
Rudolph de Roode, Experiment Station, West Virginia	1.82 1.86 1.88	1.85	.....	1.79	1.82
J. R. Harris, Experiment Station, North Carolina...	1.98 2.01	2.00	1.91	1.96	1.88
B. B. Ross, Experiment Station, Louisiana.....	1.81 1.83	1.82	.....	.....	.....
A. E. Knorr, Department of Agriculture, Washington, D. C.....	1.72 1.75	1.74	1.69	1.91	1.65
Averages		1.86	.....	1.91	1.77

[1]Without addition of NaCl. [2]HNO_3 + HCl to destroy organic matter.
[3]After ignition in H_2SO_4 dissolved in weak acid.

SAMPLE 2.—CRUDE POTASH SALTS, COTTONSEED MEAL, AND HULL ASHES.

Analysts.	Lindo-Gladding with H_2SO_4.		Lindo-Gladding water solution.	Alternate, two filtrations.	Alternate, one filtration.
	Per cent.		*Per cent.*	*Per cent.*	*Per cent.*
J. S. Meng, Experiment Station, Mississippi	5.05 5.13	5.09	.....	.....	.....
W. S. Sweetser, Experiment Station, Pennsylvania.	5.00 [1]5.03 [2]5.37	.....	.....	.....	.....
R. B. Moore, Experiment Station, Massachusetts...		4.90	.....	.....	.....
E. H. Farrington, Experiment Station, Illinois.....		5.15	.....	5.11	.....
A. L. Winton and O. W. Ogden, Experiment Station, Connecticut	4.70 4.59	4.65	[3]5.03 [3]4.99 5.01	.....	.....
F. W. Morse, Experiment Station, New Hampshire.		4.77	.....	5.13	4.93
Rudolph de Roode, Experiment Station, West Virginia		4.95	.....	4.89	4.92
J. R. Harris, Experiment Station, North Carolina ..	5.18 5.14	5.16	5.03	5.04	5.11
B. B. Ross, Experiment Station, Louisiana	4.52 4.60	4.56	.....	.....	.....
A. E. Knorr, Department of Agriculture, Washington, D. C	4.84 4.85	4.85	4.81	5.00	4.83
Averages		4.91	.....	5.03	4.95

[1]Without addition of NaCl. [2]HNO_3 + HCl to destroy organic matter.
[3]After ignition in H_2SO_4 dissolved in weak acid.

SAMPLE 3.—COMMERCIAL SULPHATE OF POTASH.

Analysts.	Lindo-Gladding.		Alternate, two filtrations.	Alternate, one filtration.
	Per cent.		*Per cent.*	*Per cent.*
J. S. Meng, Experiment Station, Mississippi	50.69 50.86	50.77		
W. S. Sweeter, Experiment Station, Pennsylvania	51.18 [1]51.26	.....		
R. B. Moore, Experiment Station, Massachusetts		50.56		
E. H. Farrington, Experiment Station, Illinois		51.34		
A. L. Winton and O. W. Ogden, Experiment Station, Connecticut	50.60 50.55	50.58		
Rudolph de Roode, Experiment Station, West Virginia		50.13	49.98	50.02
J. R. Harris, Experiment Station, North Carolina	[2]51.16 51.20	.....	51.03	51.52
B. B. Ross, Experiment Station, Louisiana	50.28 50.47	50.37		
A. E. Knorr, Department of Agriculture, Washington, D. C.	50.24 50.02	50.13	50.20	50.48
Averages		50.69	50.42	50.60

[1] Without addition of NaCl. [2] Usual Lindo-Gladding with fertilizers.

Connecticut furnishes the following notes: The K_2PtCl_6 in every case was washed from the filtrate after weighing, with boiling water, and the filter (Gooch) dried at 100° C. and weighed. In every case it weighed somewhat more than it did originally, and a corresponding correction was made. The total correction was equivalent to from 3 to 4½ milligrams of K_2PtCl_6.

Pennsylvania sends these notes: I used strong HNO_3 plus a few drops of HCl to destroy the organic matter in the determination of total K_2O in Nos. 1 and 2, sulphuric acid not proving satisfactory. The use of HNO_3 requires less time and makes neater work. I think it is better not to add the NaCl solution. I have made a large number of comparisons, and find that the K_2PtCl_6 is much more readily washed and better results are obtained.

In sample No. 1, containing tobacco stems, muriate and sulphate of potash, the average by the Lindo-Gladding, with previous treatment with H_2SO_4 is 1.86 per cent. The highest determination is 2.20, the lowest 1.65; an extreme variation of 0.55, which is too great. This is a percentage of the whole K_2O present, equal to 29.03. In sample No. 2, where cotton-seed meal is present in addition to hull ashes and potash salts, the average is 4.91 by the Lindo-Gladding incinerating with H_2SO_4. The highest is 5.16, the lowest 4.56, an extreme variation of 0.60, which, though too great, is still much better than results recorded in sample No. 1; this extreme variation corresponds to 12.20 per cent of the total K_2O present. In sample No. 3, a commercial sulphate of potash, the Lindo-Gladding method for sulphates, kainit, etc., gave an average of 50.69. The highest is 51.20, the lowest 50.13, an extreme variation of 1.07 per cent. This is a percentage of the whole K_2O, equal to only 2.11 per cent.

In regard to the question of one filtration for the alternate method, the evidence appears to be that such a plan yields somewhat lower results than by two filtrations. Such being the case, I do not feel that we would be justified in making any change.

There are four points which I would call to the attention of the association and ask for a consideration. I do not offer them as recommendations:

(1) The possible reduction of the platinum solution to $PtCl_2$ when evaporating the last time. It is thought that if the water bath is kept at a brisk boiling, the solution on coming to dryness may sometimes be reduced to $PtCl_2$.

(2) The addition of NaCl in evaporating the last time: Some claim that this is not necessary, others say it is. Let us have some discussion in regard to it.

(3) Dissolving the final precipitate in hot water and weighing any residue to be subtracted from the original weight, in order to get the true weight of the K_2PtCl_6. This is not laid down in the method, yet some practice it now.

(4) Is H_2SO_4 sufficient for incinerating organic potash materials? One analyst suggests strong HNO_3 plus a few drops HCl in its place.

As to recommendations, I would recommend no changes from the methods of last year. I would recommend, however, two changes of arrangement:

(1) Strike out the words "pulverize the fertilizer (200 or 300 grams) in a mortar" in the second line from the bottom on page 209, Bulletin 28. This is unnecessary here, as the preparation of the sample is already provided for.

(2) All the words following "To prepare the washing solution of NH_4Cl," in section (4), p. 210, should be transferred to the conclusion of section (1), on page 210. Preparation of this reagent more properly belongs under section (1) instead of section (4).

In conclusion I would say that the results this year are not as satisfactory as they should be. Whether it is because it is considered that the existing methods can not be improved upon, and so proper attention was not given to the details of the analysis, I am unable to say. But that more care is necessary in this determination is very evident.

Mr. Winton presented the following paper:

ON THE USE OF SODIUM CHLORIDE IN THE LINDO-GLADDING METHOD OF DETERMINING POTASH.

The following extract taken from Mr. Gladding's paper read before this association September 1, 1885, explains why the use of NaCl in the Lindo-Gladding method has, been deemed necessary:[1]

To test the method applied to the estimation of potash in the form of sulphate, chemically pure K_2SO_4 was employed, using an excess of platinum solution and several cc. of HCl. The results were as follows:

	K_2SO_4 taken.	Weight of precipitate.	K_2SO_4 obtained.
	Grams.	*Grams.*	*Per cent.*
1	0.12539	0.8580	100.95
2	0.13228	0.3790	102.81
3	0.49780	1.4148	101.45

These results show that with HCl alone added the precipitate is not pure K_2PtCl_6. It may be that the presence of free H_2SO_4 causes the error. To test this an amount of NaCl about equal to the K_2SO_4 was added in the following experiments, thus avoiding the presence of free H_2SO_4:

	K_2SO_4 taken.	Weight of precipitate.	K_2SO_4 obtained.
	Grams.	*Grams.*	*Per cent.*
1	0.134977	0.3782	100.01
2	0.121427	0.3415	100.16
3	0.122500	0.3429	99.95

Without doubting the accuracy of these experiments, and the necessity of using the NaCl where the percentage of potash is high, I have long entertained a suspicion that in the analysis of ordinary fertilizers containing a low percentage of potash, the error introduced by omitting NaCl entirely would be inconsiderable.

[1] Bul. No. 7, Chem. Div. U. S. Dept. of Agr., p. 40.

An opportunity for testing this theory was presented a short time ago, when potash was determined in 24 superphosphates, with and without NaCl. The determinations with NaCl were made by Mr. Ogden, those without, on different solutions, by myself. The results are as follows:

With NaCl.	Without NaCl.	Without NaCl, + or −.	With NaCl.	Without NaCl.	Without NaCl, + or −.	With NaCl.	Without NaCl.	Without NaCl, + or −.
Per ct.	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*
5.54	5.39	−.15	1.71	1.69	−.03	4.70	4.76	+.06
3.09	3.05	−.04	5.66	5.64	−.02	3.96	3.96	.00
11.07	11.17	+.10	1.60	1.59	−.01	1.68	1.72	+.04
6.08	6.22	+.14	6.09	6.13	+.04	1.06	1.14	+.08
2.58	2.54	−.04	6.14	6.13	−.01	1.51	1.60	+.09
2.04	1.95	−.09	1.82	1.84	+.02	2.23	2.27	+.04
4.93	5.02	+.09	2.41	2.51	+.10	1.27	1.43	+.16
6.54	6.63	+.09	1.56	1.57	+.01	13.73	13.66	−.07

The analyses were performed in the usual rapid way during the rush of fertilizer work, and the differences in the results are no greater than occur in duplicate determinations when exactly the same method is employed, and are due largely, if not entirely, to analytical error.

The substantial agreement of these results led to the trial of similar experiments with the commercial potash salts ordinarily used for fertilizing purposes, viz, high-grade sulphate of potash, double sulphate of potash and magnesia, and kainit.

A water solution was prepared from a sample of each of these three salts, and a number of portions of 50 cc., each representing 0.5 gr. of the salt, were drawn off into platinum dishes and evaporated on boiling-water baths, with 2 cc. of concentrated HCl, and quantities of NaCl and platinum solution as given in the table.

Since the point to which this evaporation is carried may influence the results, particulars are here given in detail. When only a few cc. of liquid remained, the dishes were removed at short intervals from the baths, and the liquid caused to run over those portions where a residue had already deposited. This was continued until after removal from the bath, and after a few moments cooling the residue was left in a pasty condition. In no case was the evaporation carried to complete dryness. Eighty per cent alcohol was then added, and the lumps broken up with a rod. After standing at least one hour, with frequent stirring, the residue was collected on Gooch crucibles, washed thoroughly with 80 per cent alcohol, and then with two portions of 25 cc. each of NH_4Cl solution (prepared in the usual way), the first portion being run through once, the second six times. After a final washing with alcohol the K_2PtCl_6 was dried at 100° C. The results are as follows:

	High-grade sulphate of potash.	Double sulphate of potash and magnesia.	Kainit.
	Per cent.	*Per cent.*	*Per cent.*
0.5 gr. NaCl; 20 cc. Pt. Sol	50.59		
0.25 gr; NaCl; 15 cc. Pt. Sol	50.65	26.90	10.03
No NaCl:			
15 cc. Pt. Sol		27.00	9.98
10 cc. Pt. Sol	50.56		
8 cc. Pt. Sol		26.98	9.90

If the addition of NaCl is necessary, 0.25 gr. (the quantity directed for sulphate of potash) in the case of high-grade sulphate is certainly only about half enough to furnish base for combining with the H_2SO_4. The table shows, however, that the results are practically identical whether an equivalent quantity of NaCl was added to the potash solution, or whether no NaCl was added at all.

Since there was no pure K_2SO_4 at hand, Gladding's experiments could not be repeated with a chemically pure sulplate, although the results obtained on the high-grade commercial sulphate would be expected to show variations similar to those obtained using the pure salt.

The following determinations with pure KCl were designed to show the influence of free HCl and H_2SO_4. The method employed was the same as that already described, except that in each case 10 cc. of platinum solution was used, and in no case was the precipitate washed with NH_4Cl solution. The results are as follows:

	Conc. HCl taken.	H_2SO_4 taken.	KCl taken.	K_2PtCl_6 obtained.	KCl obtained.	KCl obtained.	K_2O obtained.
	CC.	*Grams.*	*Grams.*	*Grams.*	*Grams.*	*Per cent.*	*Per cent.*
1	none	none	.4679	1.5315	.4678	99.99	63.18
2	none	none	.4789	1.5680	.4791	100.04	63.22
3	2	none	.4717	1.5385	.4701	99.67	62.98
4	2	none	.4125	1.3475	.4118	99.83	63.08
5	2	.35	.4325	1.4105	.4310	99.66	62.97
6	2	.50	.4958	1.6220	.4956	99.96	63.17
7	2	.75	.4700	1.5363	.4694	99.89	63.12
8	2	1.00	.4669	1.5251	.4660	99.81	63.07
Theory							63.19

These figures show that the presence of free HCl, or HCl with 1 gram or less of H_2SO_4 exerts but little if any influence on the results. Experiments 5 to 8 in the table are practically equivalent to experiments with chemically pure sulphate to which an excess of sulphuric acid has been added. There seems to be a slight tendency toward lower results when H_2SO_4 is present, but the greatest difference in any case was but 0.22 per cent of K_2O, while Gladding obtained results on K_2SO_4 in each case much higher than the theory. If the addition of NaCl is unnecessary, the advantages of its omission are obvious:

(1) The trouble of making up and using one reagent would be dispensed with;

(2) A saving of platinum solution would be effected.

In the case of kainit and double sulphate it will be noticed that a half quantity of platinum solution was found to be sufficient. In the analysis of superphosphates the quantity could be also proportionately reduced. At the present high price of platinum this is a point of no small importance;

(3) The time of digestion of the residue with alcohol after evaporation with platinum solution might be considerably shortened, since the presence of a large amount of Na_2PtCl_6 would be avoided.

The attention of this association has bean called to the foregoing experiments with the hope that others will be induced to investigate further the points in question.

Mr. Terne presented the following paper

ON SOURCES OF ERRORS IN THE DETERMINATION OF POTASH.

In the early part of spring we had a case of potash determination on hand which caused a great deal of trouble to our State chemist, Prof. Frear, as well as to myself.

The goods were a mixture of 1,600 pounds of ammoniated phosphates and 400 pounds of muriate of potash. The guarantee was calculated on the analysis of the two com-

pounds to be: 10 per cent K_2O, 6 per cent available phosphoric acid, 2 per cent ammonia. This mixture fell in the hands of Prof. Frear, who made the following analysis:

Moisture	11.58
Soluble phosphoric acid	5.05
Reverted phosphoric acid	1.31
Total available phosphoric acid	6.36
Nitrogen	1.92 = 2.77 ammonia.
Potash	8.66

The last item was 1.34 per cent below guarantee. As every other item of the professor's report corroborated our own work, and I was positive that the above-named quantity of muriate of potash had been used, I remonstrated against the correctness of the potash determination. Very shortly after this Prof. Frear sent us a synopsis of analyses of the same sample, all confirming his first report. We had no doubt as to the correctness of Prof. Frear's work, but knowing that the correct quantities of muriate of potash had been used, we questioned whether the samples taken rightly represented our goods.

I had the same mixture made for stock over again, and gave the strictest orders for accurate weighing and mixing. I am perfectly satisfied that all was done to insure a uniform mixture, that possibly could be done in the way of manufacturing. The sample sent to the laboratory gave, to my astonishment, potash 8.93 per cent. I took a second sample, and to my disgust I found potash 8.49 per cent. We made another mixing; result, potash 8.88 per cent. The muriate of potash was warranted 50 per cent K_2O, and though it was never known to me to fall below standard, I had the stock of muriate of potash re-examined.

The muriate of potash used in this mixing contained 50.95 per cent of $K_2O = 80.52$ per cent of muriate of potash, which, according to this analysis, should give in the mixing 10.13 per cent K_2O. Seemingly standing before a conundrum, I had a third mixing made with the same care as before, but instead of running the mixtures twice over the screen, as we always do, I had this mixing run over four times.

The results in two different samples from this pile now were:

	Per cent.
K_2O	10.15
K_2O	10.09

Complete fertilizers are all mechanical mixtures, and it can be demonstrated to any mind forcibly and clearly that perfect mixing is not possible even with the best of machinery. Accurate weighing and greatest care in handling are required to make the goods so uniform that the small samples taken for analysis shall always represent the goods fairly, and come up to the guarantee based on the analysis of the ingredients of the mixture.

Now in our case, a mixture of 2,000 pounds contains 400 pounds of muriate of potash. The method of analysis requires a sample of 10 grs., which should contain 2 grs. of muriate of potash. But if the sample should contain 0.2 gr. of muriate of potash less, or only 1.80 gr., your analysis will give you only 9 per cent. Or if another 0.1 gr. should be missing, this difference will give the analytical results of Prof. Frear and myself. Now, gentlemen, do you think it possible to avoid such occurrences? In my twenty years' practice in the fertilizing trade of the United States I know of no case which demonstrates more forcibly the utter impossibility to judge the composition of a brand of mixed fertilizers by a single analysis. I lay this case before you to show you the injustice that such discrepancies between the guarantee and the analysis may work, making our goods appear unsatisfactory.

Your work in the laboratory, on the samples on hand, may be perfectly correct and still do injustice to the goods. If you have confidence in your work do not waste time on duplicate analyses, but take another sample of the same brand. The difficulty of complete mechanical mixing increases with the lowering of K_2O to such a degree, that I take particular care in mixing the goods for a long time in cases where the guarantee is as low as 2 per cent of potash, using the 80 per cent muriate of potash,

100 pounds per ton of which should really give 2.5 per cent of K_2O. Goods which analyze 2 per cent of ammonia, 8 per cent of available phosphoric acid, and 2 per cent of potash, or something similar to this, are much in demand just now. Your sample of 10 grs. of such goods would then contain 0.5 gr. of muriate of potash. If this sample fall short in muriate of potash by only 0.05 gr., your analysis will show a deficiency of 0.2 per cent of K_2O. The house which I have had the honor to be connected with since 1876 prides itself on the constant control exercised over the raw material and the factory products for use as a basis for future mixings, on the care taken in securing mechanical uniformity, and on the employment of all possible means to keep the goods up to the warranted standard. But, as demonstrated by the potash determinations referred to above, it is a matter of absolute impossibility to manufacture mixed fertilizers of a character as uniform as you could make chemical products. Add to the insufficiency of the mechanical mixing of samples the frequent errors of laboratory work (for instance, as 0.001 gr. K_2PtCl_6=0.038 per cent K_2O, an error of 0.003 gr. K_2PtCl_6 in the analysis will make a difference amounting to 0.114 per cent of K_2O), and it is really astonishing that the general results of the analytical work are so satisfactory to both the analyst and the manufacturer. No other ingredient of fertilizers causes as much trouble in its determination as the potash.

Finally, I repeat my request to the gentlemen in control of fertilizer analysis not to be satisfied with the analysis of one sample, but, before condemning the brand of a reputable house, to make at least a duplicate on an entirely new sample of the brand under investigation.

Mr. BARTLETT. I have no paper to present, but want to say a word about the method of one filtration. Data obtained by that method indicate that it will give as high results, and in many cases a little higher, than either the Gladding, or the old method of two filtrations. A few years previous I made fifty determinations, with one and two filtrations, and the results agreed very closely, not differing more than two or three tenths of one per cent. Mr. Merrill, my associate, has recently made six determinations in comparison with the Gladding method. Four gave higher results by the method of one filtration than by the Gladding, but in no case over two tenths. One of the others was the same as, and one was slightly lower than, the Gladding. I used the method in a slightly different manner from that given in the report. I used no barium chloride or oxalate of ammonia. I simply used barium hydrate, usually adding quite a quantity to the solution before it is made up to the amount from which the aliquot part is taken, in that way avoiding quite a quantity of precipitate in the final filtration. After the aliquot part is taken I add enough barium hydrate to make the solution alkaline, heat for a short time on the steam bath, remove it from the steam bath, and then add enough ammonium carbonate to precipitate the excess of barium, and filter immediately, and in that way get very good results. I prefer this method as being more rapid than the Gladding.

Mr. FREAR. I recall a result I reported last year of similar work done by Mr. Sweetser, one of my associates. On more than twenty-five samples of fertilizers containing potash he got practically the same results with and without the use of sodium chloride.

Mr. HUSTON. I desire to call attention to the subject of the condition of the potash when finally weighed, brought up in the association a num-

ber of years previously. I have made a great many observations in regard to the double salt when weighed, and with the utmost care have never been able to get a good clean precipitate in working with the commercial goods with which I have to deal. I use the Lindo-Gladding method, washing with chloride of ammonium solution as directed in liberal quantities, but there is always some dirt on the filter; I have, therefore, abandoned the use of the asbestos filter, and now filter on ordinary filter paper, afterward dissolving in hot water and evaporating to dryness. The goods I have to use are largely bone and tankage material, and it is almost impossible to get a thoroughly clean precipitate. I have been very much pleased with drying in platinum dishes weighing about 50 grs., which I have used over and over again; and while there was objection always to long evaporations, yet in a series of determinations not much time is lost, and I have been very much surprised at times at the large amount of insoluble matter on the filter, even when the solution, before it was precipitated, appeared to be very clear and clean. I know the fault is not with the platinum solution, because that has been made up in the laboratory, and has been prepared with great care.

Mr. DE ROODE. I have had the same experience as Mr. Huston, but instead of evaporating the solution after dissolving and weighing, I generally filter the precipitate on a known weight Gooch crucible, then weigh the crucible.

The two recommendations of the reporter, namely, to strike out the words "pulverize the fertilizer (200 or 300 gr.) in a mortar," on page 209, two lines from the bottom, and on page 210, to transfer the method for preparing the ammonium chloride solution from section 4 to section 1, were then taken up and adopted, and the convention adjourned until Saturday morning at 9:30 o'clock.

THIRD DAY.

MORNING SESSION, SATURDAY.

The convention was called to order by the president at 9:30 o'clock.

At the request of Mr. Myers, who had not been present the previous day, Mr. McDonnell moved that the association reconsider its action in striking out the method of standardizing by potassium tetra-oxalate. Carried.

Mr. MYERS. I move that potassium tetra-oxalate be allowed to stand as one of the alternate methods for standardizing the solution in the nitrogen determination.

Mr. JENKINS. I have found that this potassium tetra-oxalate had, in the hands of some of my assistants, given very sharp and very excellent results, and I think that anything that will give a sharp result to chemists working skillfully ought to be retained, as it is a great advantage to standardize by a number of entirely distinct compounds.

Mr. VAN SLYKE. I have found the potassium tetra-oxalates method very satisfactory, and the action of yesterday, in striking it out, I think rather hasty. I do not think it is moving in the right direction to lessen the number of methods, but there should be a greater number if good ones could be suggested.

Mr. PAYNE. I call attention to the fact that the official methods indicate the use of ammonia altogether as an alkali, and the tetra-oxalate will not work well with ammonia. If the gentleman will include soda in addition to the ammonia, I would be willing to vote for its retention.

Mr. MYERS. I have made the motion because I dislike to see the association get too narrow in its methods of standardizing solutions. I like to see a chemist have some liberty of action, and be able to avail himself of the researches and experience of the best laboratories in the country. Including the tetra-oxalate as an alternate method will not compel any one to use it against his wishes.

Mr. FREAR. I voted to eliminate the tetra-oxalate method because it has not given satisfactory results in my work, and while I think the method well worth a trial, I do not think it wise to adopt as an official method one which has been proved not to be satisfactory in the hands of a large number of chemists of fair ability. Certainly there is nothing to prevent any chemist from using the tetra-oxalate as a check.

Mr. COOKE. It is not necessary that the same alkali should be used in standardizing the acid as is to be used afterward in the actual work. I have used tetra-oxalate in connection with the other methods and like it very much, and will be sorry to see it struck out.

Mr. MCDONNELL. I have found in my experience that different indicators, as suggested by the last speaker, can not be used indiscriminately. I have found as much difference as 0.5 cc. in 20 cc. of one-tenth normal acid in the use of different indicators, such as cochineal and phenol-phthalein.

Mr. COOKE. When that happens there must be something wrong, because if the indicators are correct the same results should be obtained.

Mr. DE ROODE. The trouble is not with the indicators and the solutions used, but in not being able to get the tetra-oxalate pure. I have never been able to get it pure.

The PRESIDENT. I have always been able to get the substance pure when I make it myself, and am friendly to its use, but do not think it ought to be adopted in face of the opposition.

Mr. SCOVELL. I have not made it myself, but Prof. Peter has found no trouble in making it pure. The only difficulty in its use is in getting an indicator with ammonia; we have used soda for the alkali with satisfactory results. I am in favor of retaining the method for another year.

Mr. JENKINS. I do not think there would be any difficulty in preparing pure tetra-oxalate if one would purify his own oxalic acid. Of course

it could not be made with the oxalic acid that could be bought, because that is never pure.

The motion was then put and carried.

Mr. VAN SLYKE. I move, in this connection, that the reporter on nitrogen for next year be requested to pay special attention to experimenting with tetra-oxalate and indicators, and to methods of standardizing in general, as far as possible. Carried.

The consideration of potash was then resumed, and Mr. McDonnell moved that the use of sodium chloride in the Lindo-Gladding method be dismissed.

Mr. PAYNE. I move, as a substitute, that action on that be suspended until it has been tried. It is the intention that the next reporter on that subject is to have it thoroughly investigated, and it would seem better to have the full sense of the different members of the association before making any change in that analysis. I think there are a good many who have not tried it, and who would like to feel sure before voting either way. Carried.

Mr. KILGORE. I beg to refer to the directions on page 210 in regard to the residue being taken up with enough water to dissolve the double salt. I can not see the use of that, and move that it be struck out, and that 80 per cent alcohol be used.

Mr. PAYNE. I call the attention of the last speaker to the fact that the method only mentions alcohol, not 80 per cent alcohol. If it were made to say 80 per cent alcohol it would be all right. My custom has been to add enough water to dissolve the chloride. If no water were used, the use of 95 per cent alcohol would cause the analyst to have great trouble in getting rid of the ammonium chloride without a great deal of washing. I think it ought to be changed, and that the direction should be to use 80 per cent alcohol, and that enough water should be added to dissolve the platinic chloride.

Mr. JENKINS. I remember that this matter came up several years before, especially as to the extent to which evaporation should be carried. The idea in evaporating to dryness has been to get rid of the hydrochloric acid. The idea is that by evaporating to dryness all the free acid is got rid of; then by the addition of a little water, just enough to hydrate the salts, the sodium platinum salt is made more soluble in alcohol. I do not believe this is necessary. If sufficient were added to dissolve it, a great deal would be required. I think that *sufficient to dissolve it* ought to be struck out, leaving the direction simply to evaporate to dryness.

Mr. PATRICK. I have done a great deal of work in analyzing fertilizers in past years, and my experience has been that the dry residue of platinum chloride should be moistened with water and made up in a thick or thin paste; otherwise it would take a long time to dissolve. I will therefore offer as a substitute for the motion before the house, that

it be made to read that the residue be moistened with water and mixed up to a thin paste with strong alcohol.

Mr. KILGORE. I think if it were possible to get at the exact quantity of water added it would be all right, but if the analyst makes up his alcohol solution to 80 per cent he will know just what to do. I think 80 per cent alcohol would be more accurate.

Mr. LORD. I coincide with this view. It has been my habit to evaporate to dryness, add 80 per cent alcohol, and let stand until dissolved; and I never have any trouble in getting a clean precipitate. I do not see why a small quantity of water should be used. By adding 80 per cent alcohol enough water is added to effect the solution.

Mr. Patrick's motion was put and lost.

The PRESIDENT. The original motion is before the house, and is, as I understand it, to strike out the words, "taken up with a little water, sufficient to dissolve it," and insert instead, "treat with 80 per cent alcohol."

Mr. DE ROODE. I think the Lindo-Gladding and the alternate methods should be the same in this respect. The alternate method reads "Now evaporate on the water bath to a thick sirup." I think this would be better, because if evaporation were carried to dryness, especially if the water in the bath were boiling, there would be a liability of loss of platinum. I move to amend the motion so that in the Lindo-Gladding method the treatment is the same as in the alternate method so far as evaporation is concerned.

Mr. LORD. I suggest an objection to that. If the direction requires evaporation to a thick sirup, the matter as to what constitutes a thick sirup is left to the judgment of the analyst. I think evaporation should be carried to dryness; if done with care and at not too high a temperature, reduction is not to be feared; and then 80 per cent alcohol should be added. The solvent action of that liquid is known, and the element of uncertainty would thus be eliminated.

Mr. PATRICK. How long will it take for the 80 per cent alcohol to perform its work?

Mr. LORD. I have never kept a time record. As a rule I filter and wash the precipitates from twenty minutes to an hour after treatment.

Mr. PATRICK. I think that, inasmuch as the analyst is not always sure how much double sodium salt there is in an unknown substance, he will not know whether to let it stand twenty minutes or an hour after treatment with 80 per cent alcohol, and I still believe the method proposed by myself is best.

Mr. BATTLE. It is best to evaporate to a thick paste, because in that event the trouble of possible reduction is avoided.

Mr. DE ROODE. I always perform this operation by evaporating to a pasty condition, then when the dish is removed from the water bath and the thick sirup allowed to roll around the dish, it will solidify on

cooling. I do not see that the little water there could possibly make any difference.

The amendment was then adopted, as was also the original motion as amended.

Mr. PAYNE. I think it will be well to insert in the method for potash determinations some caution as to the presence of ammonia during evaporation. In the sample sent out by Mr. Battle I made a number of determinations, but on account of the legislature convening about that time, I was obliged to leave the evaporated salts in the dishes for some days. Afterward, on making the determinations, I found that the results agreed very closely with the general average given in the report; but I did not report them, because I was convinced they were too high. I put out a blank also with platinic chloride to see what effect the ammonia would have, and had such an amount of precipitate that I am sure the determinations were all too high. I move that a caution in regard to the exposure of potash solutions to ammonia, especially during evaporation, be inserted in the method. Carried.

(See Mr. Battle's Memorandum of Changes, p. 148.)

Mr. DE ROODE. I move that in the alternate method, page 211, the words "or further" be struck out, and in place of "strong alcohol" "80 per cent alcohol" be inserted. Carried.

Mr. BATTLE. From the reports received, some analysts think sulphuric acid not sufficient for getting rid of the organic matters in fertilizers containing potash in an organic state. Some suggest the use of nitric and hydrochloric acid. I ask for the views of the members of the association on this point.

Mr. DE ROODE. I have found that after incineration and ignition with sulphuric acid the mass will stick to the dish, so that it is almost impossible to get it off, but if a little hydrochloric acid be added and it is warmed a little, it will loosen itself and come off perfectly.

Mr. ROSS. I have found it necessary to transfer to a mortar and rub up thoroughly to a homogeneous paste with a little water before boiling; otherwise it is found almost impossible to effect complete solution.

Mr. DE ROODE. I move that, where this organic matter has to be destroyed with sulphuric acid and ignition, strong hydrochloric acid be added so as just to moisten the mass, which should then be slightly warmed so as to loosen it in the dish. Carried.

Mr. ROSS. I desire to call attention to the addition of 5 cc. of sodium chloride solution, page 210, and think this entails a useless amount of liquid to be evaporated. I always add the sodium chloride in a solid state, approximately a tenth of a gr. instead of 5 cc. in solution.

Mr. HUSTON. I move that the words "or its equivalent" be added there. Carried.

Mr. BATTLE. I have one other point I want to mention. Some of the analysts in the different States, after getting the final precipitate,

dissolve it in water and take account of any residue left by subtracting from the first precipitate, to get the total. Is this necessary?

Mr. HUSTON. I consider that it is necessary, particularly in cases where the material contains a good deal of such substances as tankage. In such cases there is always a relatively dirty precipitate, even with the best of care, and there may be as much as 1.5 per cent of the K_2O, which I think too large a fraction of the total amount to be disregarded. I therefore prefer to filter the final precipitate and weigh only what is thoroughly and clearly soluble in water.

Mr. MCDONNELL. I move to insert "with care to insure the perfect solubility of the double salts." Carried.

Mr. CARPENTER. I do not think the first sentence in section 3, page 210, should be followed exactly. It would be better to make up the solution as in section 1, except that 5 grs. should be taken instead of 10 grs.

After some discussion as to the form in which this should be expressed, the president suggested that the matter be referred to a committee, who should agree upon the amendment of this section and report back to the meeting before adjournment.

On motion of Mr. De Roode this action was taken. The committee subsequently reported an amendment to section 3 of the Lindo-Gladding method, page 210, Bulletin 28. This will be found in methods of analysis.

The president at this point stated that the president-elect had made his selections of reporters for the next year, and suggested it would be well for them to be announced at this point.

Mr. Lupton announced them, as follows:

Ash and soil.—Mr. Harrington.
Phosphoric acid.—Mr. Lord.
Potash.—Mr. Payne.
Nitrogen.—Mr. Van Slyke.
Dairy products.—Mr. Babcock.
Foods and feeding stuffs low in carbohydrates.—Mr. Anderson.
Foods and feeding stuffs high in carbohydrates.—Mr. Jordan.
Fermented liquors.—Mr. Crampton.
Sugar.—Mr. Ross.

MISCELLANEOUS BUSINESS.

No motion being under consideration, the president announced that the next order of business was the presentation of miscellaneous papers, and Mr. Battle presented the following papers:[1]

[1] The secretary has endeavored to collect under the head of miscellaneous business all the proceedings not directly connected with the reporters' work, without reference to time of consideration.

ON THE LOSS OF MOISTURE IN BOTTLED FERTILIZER SAMPLES WHEN CLOSED WITH CORK.

By H. B. Battle.

I desire to call the attention of the association to a practical difficulty which at one time worried me no little, until our system of bottling was changed some years back as the result of this investigation.

Frequently having occasion to repeat a determination of total phosphoric acid in a commercial fertilizer, I almost invariably found the second determination, made two or three weeks subsequent to the first, to give a higher result. This variation being so constant, I was forced to think that it was not the result of error of analysis, but owing to loss of moisture in the sample.

The samples of fertilizers were usually placed in a wide-mouth bottle of about two ounces capacity, and closed with an ordinary cork. It was customary to make five distinct weighings from each bottle, which was unavoidably kept open during the process of each weighing, the bottle in the mean time being subjected to the ordinary temperature of the laboratory.

To test the theory of loss in moisture, I selected 6 samples of ammoniated fertilizers entirely at random, from a lot of 30 or 40. The first determination of moisture was made one hour after sampling, and the second after the lapse of one month, with the results given below:

Table I.—*Moisture at 100° C.*

	One hour after sampling.	One month later.
	Per cent.	*Per cent.*
1	18.64	17.83
2	17.42	15.73
3	20.12	19.40
4	16.15	15.94
5	12.03	10.70
6	16.38	15.46

Table II.—*Decrease of moisture.*

	Per cent.
No. 1 decreased in moisture in thirty days	0.81
No. 2 decreased in moisture in thirty days	1.79
No. 3 decreased in moisture in thirty days	.72
No. 4 decreased in moisture in thirty days	.21
No. 5 decreased in moisture in thirty days	1.33
No. 6 decreased in moisture in thirty days	1.02

Table II shows an invariable decrease in moisture, which has the effect of elevating the per cent of the other constituents to a marked degree. For example, No. 2, where the decrease is greatest, the sample contained at the time of the first weighing 9.77 per cent total phosphoric acid. Theoretically this would be raised at the second weighing, on account of the loss of 1.79 per cent of moisture, to 9.98 per cent; an increase of 0.21 per cent of total P_2O_5. Practically this increase was found to exist, by actual analysis, thirty days after the first determination.

As a remedy to prevent this loss of moisture and consequent disarrangement of 2 sets of determinations, the use of tightly fitting rubber stoppers is recommended; even then a slight loss is liable to occur from the frequency of the weighings and exposure to air. The necessity of fitting in the rubber stoppers tightly after each weighing will be observed.

THE EFFECT OF FINER GRINDING IN THE PREPARATION OF FERTILIZER SAMPLES.

By H. B. Battle.

In the official methods of the present time, and in fact the same may be said of all the years previously, it seems to me that one of the most important operations has received the least consideration. I refer to the preparation of the sample. All that is required now is, to quote the exact words of the official methods:

"*Preparation of sample.*—The sample should be well intermixed and properly prepared, and passed through a sieve having circular perforations one-twenty-fifth of an inch in diameter, so that separate portions shall accurately represent the substance under examination, without loss or gain of moisture."

Nothing definite is said as to how fine the material is to be ground. It is definitely stated that the particles shall not be coarser than one-twenty-fifth inch in diameter, but how much finer no one seems to care. Is this definite enough? I think not. The sample shall be "properly prepared," but the question arises, what does this mean? In what way is it to be done? Shall the sample be ground in a mortar, or shall it be passed through a drug mill, and thus ground to pass the circular holes one-twenty-fifth inch in diameter?

By merely grinding in a mortar, particles, especially the organic portions of the fertilizer, are not broken apart, but only pressed into an elongated form, some of which will then pass through the sieve, although they may be fully one-fourth inch long. Such elongated particles mix with other material with much difficulty, and very easily become separated, not only by reason of their light weight, but also on account of their form. If not very carefully watched, therefore, these materials will soon find their way to the top, and tend to yield results different from the true average analysis.

On the other hand, too fine grinding of fertilizer samples will also give erroneous results, in a way which I propose to show in the following paper, which I had occasion to write some time back.

That a sample of commercial fertilizer should be finely ground for analysis is not to be doubted. The necessity is more apparent in an ammoniated guano than in an acid phosphate, for with the former, where three or four ingredients enter into its constitution, and, too, with properties so entirely different, it is extremely difficult to obtain a fair and impartial sample, unless these constituents are finely ground and carefully mixed.

In a chemical analysis where the amount used is small, only 2 gr. and less in the more important determinations, all the accuracy possible is needed to procure a result that will represent the whole mass. It is customary that the sample shall be ground so as to pass a sieve with holes one-twenty-fifth inch in diameter. The meaning of this is not that each particle is one twenty-fifth inch in diameter, for possibly one-half of the sample may pass in turn a 40-mesh, and some even a 60-mesh seive, without further grinding, but that none of the particles are coarser than one-twenty-fifth inch in diameter. In an ammoniated fertilizer the nitrogenous materials, as horn or hoof, being tougher, are disintegrated and pulverized with most difficulty, while the phosphatic constituents, with particles even harder, are ground much finer, friction between the particles themselves assisting in the action. If such a fertilizer, after being ground to pass a 20-mesh sieve and bottled, is even slightly shaken, the finer particles of phosphate settle to the bottom, leaving the coarser nitrogenous mass at the surface, and a sample from the top will give a result to some extent at variance with that nearer the bottom. This settling is the more noticeable when the moisture is not present in a large amount. Finer pulverization, then, is absolutely necessary to secure the best results. But besides giving a more uniform mass, there are other results connected with finer grinding.

For comparison both acid phosphates and ammoniated fertilizers were taken, and each sample, after being passed through sieves of 20, 40, 60, and 100 meshes to the linear inch, were analyzed.

A. Is a high-grade acid phosphate;

B. A medium-grade acid phosphate;

C. An ammoniated guano;

D. Ammoniated guano, with an extremely high per cent of insoluble phosphoric acid;

E. Ammoniated guano, which illustrated in the 20-mesh sample, in a marked degree, what has been before stated concerning the settling of the finer particles.

TABLE I.

	No. of meshes.	H_2O.	Decrease in H_2O from 20 mesh.	Sol. P_2O_5.	Insol. P_2O_5.	Ins. P_2O_5 calculated water-free sub.	Decrease in ins. P_2O_5 from 20 mesh (from previous calculations).
		Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
A	20	15.04	4.99	11.86	1.28	1.50	.48
	60	10.05		12.32	.92	1.02	
B	20	15.68	3.79	8.16	3.77	4.46	.22
	60	11.89		8.49	3.74	4.24	
C	20	12.83	1.11	5.35	1.62	1.86	.21
	40	11.72		5.72	1.46	1.65	
D	20	11.46	.....	5.07	7.15	8.07	.....
	40	.....	.....	4.98	7.10	.....	.....
	60	.....	.....	5.08	7.36	.....	.....
	100	8.28	3.18	5.05	7.29	7.95	.12
E	20	10.81	.....	3.95	3.93	4.40	.....
	40	11.06	.....	3.81	3.74	4.25	.15
	60	11.28	.....	3.75	3.30	3.72	.68
	100	9.96	.85	3.66	3.07	3.40	1.00

We see at once two noteworthy results attended with the pulverization, viz: (1) the loss of water, and (2) the decrease of the insoluble P_2O_5. The reason for the former is obvious; the latter is no doubt owing to the greater solvent action of the ammonium citrate used on the particles which are more highly comminuted due to pulverization. The loss of moisture has the effect of increasing the content of the other substances, a mere withdrawal of water, of course, raising other constituents, which is the case with the total P_2O_5, total potash, etc., and would be so with the insoluble P_2O_5 were it not for the easier solution of the finer than the coarser particles. This decrease of insoluble P_2O_5 is enough to counteract the increase caused by the loss of water, and causes a still further decrease, amounting in extreme cases to nearly 25 per cent of the total amount of insoluble P_2O_5 present in the original sample.

I will add that the loss of water in A and B is rather excessive, part of which being due to slight drying, as the sample was too wet to pass the fine sieve.

It will be seen that the decrease of insoluble P_2O_5 in D is very slight, only 0.12 per cent from 20 to 100 mesh. I was at a loss to understand this remarkable deviation from the other results, until after the determinations were completed, when I found from the manufacturer that the basis of the phosphate was a natural guano instead of a manipulated superphosphate. The reason then was plain; ammonium citrate solution acts but little quicker on fine than coarser particles of such raw phosphate.

For the sake of comparison other results are calculated and given in Table II.

TABLE II.

	No. of meshes.	Total P_2O_5.	Rev. P_2O_5.	Avail. P_2O_5.
		Per cent.	*Per cent*	*Per cent.*
A	20	14.98	1.84	13.70
	60	15.76	2.52	14.84
B	20	14.74	2.52	10.97
	60	15.32	3.09	11.58
C	20	8.92	1.95	7.30
	40	9.02	1.84	7.56
D	20	13.50	1.28	6.35
	40	.....	.....	.....
	60	.....	.....	.....
	100	13.93	1.59	6.64
E	20	10.04	2.16	6.11
	40	10.02	2.47	6.28
	60	10.00	2.95	6.70
	100	10.13	3.40	7.06

From the last table we see that incident to the loss of water and decrease of insoluble P_2O_5, there is an increase of total phosphoric acid, amounting in some cases to nearly 1 per cent.

To summarize, then, we have for the result of pulverization for fertilizer samples:

(1) The more perfect mixing of the separate ingredients, and the consequent susceptibility to better and fairer sampling.

(2) The loss of moisture, due partly to the vaporizing of water by heat generated by grinding, and partly to the action of the air on the finer particles necessarily exposed during the pulverization.

(3) The decrease in content of insoluble phosphoric acid, caused by the more complete action of the solvent on the finer particles.

(4) The increase of the total phosphoric acid caused by the loss of moisture.

(5) The increase of the reverted and available phosphoric acid, due first to the increase of the total, and second to the decrease of the insoluble.

The last four results are shown to be directly proportional to the fineness of the grinding.

In conclusion, I would suggest the advisability of careful investigation of the sample as it is being prepared. If the organic particles are dry and do not crush readily, I would suggest passing them through a drug mill before sifting, but give them only a coarse crushing. I do not like at this time to give any further specific recommendations, but only to direct the attention of our members to this most important part of the analysis.

Mr. LORD. The matter of the loss of moisture through corks is of the utmost importance. The ordinary corks procurable at present do not seem to prevent the loss of moisture from the sample in the bottle. I have had some experience with an entirely different sample from that mentioned, but which shows this loss of moisture to a very marked degree, namely, a sample of coal. Coals when first analyzed may contain a considerable quantity of moisture, and the extreme rapidity with which the moisture is lost, even when the sample is thoroughly corked, is marked. In order to check the matter I prepared several samples of coals, and immediately determined the moisture by drying at 100° C. for one hour. Then the samples were corked up in ordi-

nary laboratory sample bottles, kept on shelves, and the moisture redetermined at intervals of a week or two for several weeks, and there was a steady and very rapid loss of moisture. The bottles were corked with what were considered to be good laboratory corks, and were only opened when the samples were taken out for test, but the loss of moisture was sufficient to have utterly vitiated the conclusions drawn from any analyses which might have been made a month after the samples had been corked. My recollection is that the heating power of those coals, calculated from the analyses which would have been made a month after its preparation, would have been about 10 per cent higher than when first prepared. I have a case also in regard to fertilizers. The sample was prepared, and the analysis made in June or July, and in November a discussion arose over the amount of total phosphoric acid, and I was called upon for a re-analysis of the sample, and the loss of moisture had been sufficient to make a perceptible but fortunately not wide difference in the proportion of total acid.

Mr. PAYNE. The corkmen designate miserable corks as fine, the next grade as extra fine, and the next as extra superfine. Ether, put in a bottle corked with the best grade of cork, and left for a very long time on a shelf, will be found to have evaporated one-half, and sometimes a bottle which has thus been standing will be found to be empty. This shows that any cork may be penetrated by vapors, but still an extra superfine long cork would retain almost anything that the bottle would, except ammonia. I have tried all sorts of experiments in attempting to keep ammonia corked up in bottles without success, and finally tried a special experiment. I sealed the bottles having new and well-ground glass stoppers with paraffine, and then covered them with rubber, wrapped the bottles up and put them away. I have recently opened them after leaving them standing for two years, and found that there was absolutely no loss of ammonia; but it was the only way I have ever succeeded in keeping ammonia from escaping from bottles.

Mr. HUSTON. Not enough is known about exact water determinations to form very important opinions on this subject. I think there is a question in some of these cases whether the water escapes, or whether the determination fails to show it. The determination of water, particularly in fertilizers containing superphosphates and organic matter added for supplying nitrogen, is a very difficult matter, and I have considered it so useless, that for two years I have omitted it in my determinations. I consider this an important matter, and one well worthy the attention of the association.

In reference to the pulverization of the sample, I think in case of a sieve that the area of the opening or the size of the wire ought to be specified.

Mr. BATTLE. In the special instance mentioned in my paper I made the determinations myself by exactly the same method, consequently

there could not have been the difference which might have been possibly attributable to the fact that the work had been done by different analysts.

Mr. LORD. I quoted the illustration in regard to coals because they are not subject to the same variability as fertilizers. The determinations were made by the same man, and I think the results prove that there is loss of moisture to a very considerable extent through the corks.

Mr. BATTLE. I offer the following resolution:

Resolved, That the secretary of the association be requested to insert the methods for fertilizer analysis at the beginning in the record of the official methods in the annual proceedings, and arrange them in the following continuous order:

(1) Preparation of sample;
(2) Determination of moisture;
(3) Determination of phosphoric acid;
(4) Determination of nitrogen;
(5) Determination of potash.

The resolution was adopted.

The president called for the report of the committee on a method for the analysis of foods rich in starch, and Mr. Woods read the report, as follows:[1]

Mr. DE ROODE. I think it would be a good thing for the assoication to adopt some uniform system of atomic weights.

The PRESIDENT. What system is recommended?

Mr. DE ROODE. I move that the matter be referred to a committee, to look into the subject and recommend some system for adoption by the association. Carried.

The PRESIDENT. On the adoption of a system of atomic weights, Messrs. De Roode, Voorhees, and Jenkins will serve as a committee.

At 12:30 noon a recess was taken.

AFTERNOON SESSION, SATURDAY.

The convention was called to order by the president at 2 p. m.

Mr. CRAMPTON presented the report of the committee on approximate methods for the determination of alcohol and sugar in sweet wines.[2]

Mr. LUPTON. I move that the report of the committee be accepted, and the method be entered in the proceedings, but not as an official method.

Mr. PAYNE. I support Mr. Lupton's motion.

The committee did not expect the report to be adopted as an official method.

The motion was put and carried.

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[1] See section on Cattle Foods, p 63.

[2] See Methods of Analysis, p. 208.

ELECTION OF OFFICERS.

The PRESIDENT. The election of officers is now in order. On motion, Messrs. Van Slyke, Jenkins, and Woods were appointed a committee on nominations.

The committee on the nomination of officers reported their recommendations, as follows:

For president, Mr. N. T. Lupton.

For vice-president, Mr. S. M. Babcock.

For secretary, Mr. H. W. Wiley.

For executive committee, Messrs. C. L. Penny and H. A. Huston.

Mr. BATTLE. I move that the secretary be instructed to cast the ballot of the association for the gentlemen recommended by the committee. Carried.

The PRESIDENT. The ballot has been cast by the secretary, and the officers mentioned in the report have been elected for the ensuing year.

The PRESIDENT. I call for the report of the committee on a National Chemical Society.

Mr. Wiley, as chairman of the committee appointed at the last meeting of the association to consider the subject of the formation of a National Chemical Society, submitted the following report:

Mr. PRESIDENT AND GENTLEMEN: The committee appointed at the last meeting of the Association of Official Agricultural Chemists on the subject of a National Chemical Society, begs to submit the following report:

On account of the impossibility of securing a meeting of our committee during the year, the chairman thereof addressed the following letter to each member on the 14th of February, 1891:

"DEAR SIR: As chairman of the committee of the A. O. A. C. on the organization of a National Chemical Society, I beg to submit, for your consideration, a proposed plan for the federation of associations of chemists of the United States.

"This plan was adopted at an informal meeting of the chairmen of the several committees, appointed by the various chemical organizations of the country, looking to the proposed union, at the Cosmos Club, on February 7.

"As it is impracticable to call a meeting of our committee at the present time, I trust that you will give the matter your earnest consideration and return to me, with such amendments as you may see fit, the plan of federation submitted. I will then collect the various opinions, thus expressed, and will collate them, and present them as the report of our committee at the next meeting of our association.

"Respectfully,

"H. W. WILEY,
"*Chairman of Committee.*"

The circular referred to in this letter was one agreed upon at a meeting of the representatives of a number of the chemical societies held at the Cosmos Club on the 7th of February this year. The associations represented at this meeting were the American Chemical Society, the Chemical Section of the American Association for the Advancement of Science, the Washington Chemical Society, and the Association of Official Agricultural Chemists. As a result of the deliberation of this meeting the circular alluded to in the letter above was prepared. This circular is appended to this report as Exhibit A.

The plan of federation which was recommended in the circular was considered at a meeting of the Washington Chemical Society on the 14th of May, and after consid-

erable discussion it was adopted without change as a tentative method of securing the desired federation. A copy of the proposed plan of federation was also submitted to to the Sugar Chemists' Association of Louisiana. At a meeting of this association on the 9th of April the plan of federation was considered and unanimously indorsed, and Mr. G. L. Spencer was selected to represent the association in any subsequent conference that might be held.

Replies were received from all members of the committee, except Prof. Nicholson. Prof. Stubbs wrote under date of March 14:

"I return herewith the proposed plan for the federation of chemists of the United States. It entirely meets with my approval. Any changes found necessary after trial can easily be made."

Dr. Frear, under date of April 22, wrote as follows:

"I have delayed making comment upon the plan of federation communicated to me some weeks since, in order that I might have time to consider it more carefully. I have several suggestions to make, but thinking it possible that there may be extensive changes in the form presented, have made no attempt to formulate them as amendments.

(1) "I very much doubt the wisdom of making undergraduate students eligible to membership, save as the constitution of some one or more of the federate organizations may entitle them to it. I would therefore qualify the word 'student' in section 3, page 2, by the prefix 'graduate.' I would not object to their admission to a separate class, to be termed 'associates,' giving them most of the privileges, but not recognizing them as full-fledged chemists. I, of course, see that this plan may offer difficulties where some undergraduates already find admittance through their relation to a constituent society.

(2) "The method of collecting fees presents some difficulties, and will work unequally. Thus, take my own case, I am a member of three of the constituent societies; the rule as it stands will require the payment of as many dollars, since no provision is made for the relief of any of the societies in such case. Again, several of the constituent organizations have no fiscal arrangements, being either branches of some larger body, as in case of the sections of the A. A. A. S., of the Franklin Institute, and of the Brooklyn Institute, or having no expenses as a body, as the A. O. A. C. This last difficulty is not insuperable, and the requirement of support from the associated societies as corporate bodies has very marked advantages. Still, it seems to me that some provision should be made for exemption where members would otherwise have to double or treble their fees.

(3) "I regard the powers granted to the council as too absolute. It is sufficiently large to throw the work into the hands of a few. The president, secretaries, and editor, with one or two others, will soon run everything. To this I would have no objection, so long as they followed closely the wishes of the majority of fellows; but human experience does not warrant us in hoping for anything so humble in their natures. I would therefore suggest that, while the power of election should remain as proposed, the tenure should be formally fixed, without precluding reëlection, if it should seem desirable, as it certainly would in several offices; and the time of election should be the annual meeting, except for temporary appointments *ad interim*. Otherwise, since the constitution places no limitation on their action, a few might call a meeting at a time inconvenient for the majority, and rush in a few of their cronies, or reëlect themselves.

"Beyond these suggestions I am very well pleased with the plan. I am glad the entering wedge for the Journal has been so adroitly slipped in by providing an editor, to whom the constitution assigns no duties.

"May I ask, without imposing too much on your busy moments, the names of the organizations represented at the meeting of chairmen on February 7, when this plan was formulated?"

Under date of July 26, Prof. Caldwell replied as follows:

"I trust that you will pardon my long delay in replying to your communication of

February 14, accompanying a proposed plan for the federation of associations of chemists of the United States.

"Of course I am unwilling to give any adhesion to any plan of this kind till after I have have heard it and other plans fully discussed in the several chemical organizations to which I belong. All that I can do now is to notice such features of this plan as seem to me questionable.

"In section 3, it being stated that all members of the constituent societies are, by reason of such membership, fellows of the association, I think it should be provided for that there should be some approximately uniform standard of admission to membership in the constituent societies; not less, certainly, than what is required in section 5 for direct admission to this association.

"Also, I would not have the qualifications for direct admission so broad as to say, 'any person who is a student of chemistry, etc.' I would at least say 'who is a special and advanced student of chemistry.'

"Section 5—duties of bibliographer and editor? Does this mean that the association is to be committed to the publication of a journal, and that we are to have another set of abstracts of all other journals in addition to those which most of us have access to already? I doubt the wisdom of this.

"Section 9—'two members' of the council to be 'elected by each constituent society.' That is to say, no matter how small one society may be or how large another, to each is given the same weight in the council. Important as are the duties assigned to the council, it seems to me that each constituent society should have an influence and weight in the council in some measure proportionate to the number of its members.

"Section 10—what is it proposed to do with the money received in payment of dues? The second part of the section seems to me uncalled for. It will simply add $1 more for the annual dues of members of constituent societies, and in so far constitute an additional provision on abstaining from membership in any such society. Better add $1 to membership fee of this association.

"Some of these criticisms may be shown to be uncalled for when discussion of the plan comes up."

Summing up the opinions of the various members of the committee, the following points appear:

(1) The chairman of the committee and Prof. Stubbs indorse without change the proposed plan of federation. Profs. Frear and Caldwell indorse the general idea of the scheme of federation, but propose certain changes in regard to membership and other details, which would alter materially the proposed plan, but not change it so much as to have it considered as a new proposition. Finally, Prof. Nicholson has expressed no opinion on the subject.

Your committee, therefore, recommends that the proposed scheme of federation, together with the opinions of the various members of the committee, be submitted for discussion before the Association of Official Agricultural Chemists, and it further recommends that it either be adopted unchanged, or with such changes as may be necessary, as an expression of the feeling of this association in regard to the proposed federation of chemists in this country. Your committee further expresses the hope that some scheme may be devised whereby all the chemists of the country may be induced to enter one common organization having branches at such places as may be found convenient to bring together, in a manner which will permit coöperation, all the chemists of the country in one national organization.

Your committee further recommends that in this proposed federation the rights and privileges of each constituent organization be respected.

All of which is respectfully submitted.

H. W. Wiley,
Chairman.
Wm. Frear.
W. C. Stubbs.
G. C. Caldwell.
H. H. Nicholson.

EXHIBIT A.

PLAN OF FEDERATION OR ASSOCIATION.

1.

This organization shall be known as the Association of American Chemists, and is formed by the federation of the following societies:

(1) American Association for the Advancement of Science, Chemical Section.
(2) American Chemical Society.
(3) Chemical Section of the Franklin Institute.
(4) Chemical Section of the Brooklyn Institute.
(5) Association of the Official Agricultural Chemists.
(6) Chemical Society of Washington, D. C.
(7) Manufacturing Chemists' Association of the United States.
(8) Cincinnati Chemical Society.
(9) Chicago Chemical Society.
(10) Louisiana Sugar Chemists' Society.
(11) Michigan Chemical Society.

2.

The object of this association shall be the cultivation and promotion in North America of chemical science, pure and applied.

3.

The association shall be composed of fellows, honorary members, and patrons.

All members of the constituent societies are, by reason of such membership, fellows of the association.

Any person holding membership in any chemical society or chemical section of any society other than the constituent societies existing in North America, may become a fellow of the association, the council consenting.

Any person who is a student of chemistry, or has been engaged as a chemist in technical pursuits, or who has received a degree in chemistry, or who has taught chemistry in any institution of repute, and is not already a member of any chemical society, shall be eligible to fellowship, the council consenting.

Such candidates shall be elected by ballot by the council, having been previously nominated in writing by two members of the association.

Honorary members shall be persons distinguished for their attainments in chemical science, and shall not be resident in North America.

Patrons shall be persons who shall contribute not less than $1,000 to the funds of the association.

Only fellows who are members of one of the constituent societies shall be entitled to hold office in the association.

4.

The association shall hold two stated meetings annually. One, the summer meeting, shall be held in connection with the annual meeting of the American Association for the Advancement of Science, at which the annual address of the president shall be delivered and officers elected, but no scientific papers shall be presented or read at this meeting. The other, the winter meeting, shall be held in a city in which there is a constituent society, at a time to be fixed by the council. No two consecutive meetings shall be held in the same city.

The summer meeting shall be regarded as the annual meeting. Special meetings may be called by the council.

5.

The officers of this association shall be a president, vice presidents, a corresponding secretary, a recording secretary, a treasurer, a bibliographer, an editor, and a council.

6.

The president shall be nominated by the ballots of all the fellows of the association, to whom blank ballots shall have been sent three months before the annual meeting. Each fellow shall be entitled to return his ballot to the secretary of the association. These ballots shall then be canvassed by a committee appointed by the council. The three persons receiving the highest number of votes shall be considered as nominated for president, and these three shall be voted for by ballot at the annual meeting of the association, and the one receiving the highest number of votes shall be declared president for the ensuing year.

The president shall be a member *ex officio* of the council, and, during the year for which he is elected, he shall preside at the meetings of the association and of the council, and shall deliver an annual address at the expiration of his term of office.

7.

The presidents of the constituent societies shall be *ex officio* vice-presidents of the association, and the council shall designate the order in which the vice presidents shall succeed to the office of president in the absence of the president, or in case of his disability.

8.

The secretaries, the treasurer, the bibliographer, and the editor shall be elected by the council; they shall be *ex officio* members of the council.

9.

The council shall be composed of the officers of the association and two members elected by each constituent society, and said members shall be elected annually by the several societies at their annual meetings.

The council shall have charge of the current business of the association and all the duties pertaining to the organization and preparation for the meetings of the association, including the election of all the officers, with the exception of the president and vice-presidents, and shall have the appointment of all necessary committees.

10.

The fellows who are not members of constituent societies shall pay the sum of $3 annually as dues. Each constituent society shall pay $1 for each member thereof in good and regular standing.

11.

The admission of new societies to the association shall be by vote of the council.

Mr. LUPTON. I suggest that the report be considered item by item. Has provision been made for a meeting in connection with the scientific association to meet in Washington next week?

Mr. WILEY. It will be proper for the association to make provision for that. The meeting referred to was called for the purpose of consid-

ering this subject, and the association should by all means be represented in that meeting. In relation to the report of the committee, I will say it is a tentative report, and I concur heartily in the criticisms of Dr. Frear and Prof. Caldwell in regard to membership. I am in favor of the formation of an American Society of Chemists. The German Chemical Society has several thousand members; the English Chemical Society has a very large membership, as also the French Society; and there ought to be a National Society of Chemists in America which would be respectable, not only in the ability, but also in the number, of its members.

Mr. VAN SLYKE. I move that the present committee be continued, and be asked to represent the association at the meeting next week, and then there will be something definite by which to be guided at the next meeting. Carried.

Mr. LUPTON. I move that the plan of federation submitted by the committee be taken up and considered item by item. Lost.

AMENDMENT OF CONSTITUTION.

Mr. FREAR. I move that the constitution be so amended as to state distinctly that only official chemists, members of the association, shall be entitled to enter motions or to vote, and ask that this matter be referred to a committee in the regular order. Carried.

The PRESIDENT. On the proposed change in the constitution, Messrs. Frear, Wiley, and Huston will please serve as a committee.

The PRESIDENT. I call for the report of the committee on a chemical exhibit at the World's Columbian Exposition.

Mr. Wiley presented the report of the committee on chemical exhibit at the World's Columbian Exposition, as follows:[1]

REPORT OF THE COMMITTEE ON A CHEMICAL EXHIBIT AT THE CHICAGO COLUMBIAN EXPOSITION.

Mr. PRESIDENT AND GENTLEMEN: The committee appointed to consider the subject of a chemical exhibit at the Chicago Columbian Exposition begs leave to submit the following report:

We see, without argument, the importance of securing at the World's Fair in 1893 an appropriate exhibit showing the equipment and methods used in chemical laboratories devoted to agricultural pursuits in the United States. In order to accomplish an exhibition of this kind, it will be necessary to enlist the active coöperation of all agricultural chemists in the country, but we are sorry to say that such coöperation is not the only element necessary to success. Equally as important is the subject of money with which to make an appropriate display, and maintain it in proper shape during the continuation of the Exposition. The suggestions which we have to submit, therefore, naturally divide themselves into two classes; first, the nature and character of the exhibit, and second the method of obtaining means to make and maintain it.

[1] The business was not transacted until the last session of the association, but is inserted here in order to have all miscellaneous business in one section.

In regard to the first subject permit us to suggest that an exhibit of a chemical nature, illustrating the relation of chemistry to agriculture, would consist in the first place of a collection of chemical apparatus suitable to special investigations on agricultural subjects. In order to secure this it appears to us that the best method would be the equipment of a complete chemical laboratory adapted to agricultural research. We do not know of any other method in which the apparatus employed in chemical research could be so effectually exhibited as in actual use. Not only should this equipment be complete from a mechanical point of view, and in view of the appliances, but it should also be supplied with competent chemists who could take charge of actual analyses relating to agricultural subjects.

As a tentative classification, we might suggest that the apparatus which should be exhibited should be of the following nature:

First, the apparatus which is particularly suited to the analysis of soils. In this case we would be compelled to trespass on the domain of our physical brethren, inasmuch as some of the most important subjects undertaken by chemists in regard to soils relate to their physical study. These investigations are so uniformly made by chemists, that all of the apparatus and processes employed properly fall within the scope of a chemical classification.

The apparatus indicated would consist of the appliances for measuring the movement of soil water, both vertically and laterally, and, in general, all the appliances connected with lysimetry. In addition to this a complete exhibition should be made of all the various appliances which are employed in separating the soil particles into classes of definite hydraulic values. All the approved methods of silt analysis should be thoroughly illustrated, and it might be well to exhibit, also, in connection therewith the various forms of apparatus which have been devised for silt analysis, even if their operation at the present time is deemed incorrect. In other words, the exhibition of chemical apparatus in this respect might easily take on something of an historical nature, showing the progress of the mechanical analysis of soils from the earlier attempts made by Nöbel and his confrères, up to the latest appliances which have been invented for that purpose. This exhibition would also naturally include all the various methods indicated above, of determining the translation of water in soils, and also the methods adopted for determining all the other physical properties so intimately connected with the chemical properties of the soil.

In regard to the chemical appliances, the methods of combustion for the determination of nitrogen, both in the wet and dry way, should be amply illustrated; also the various appliances used for the determination of humus and kindred bodies in soils. Naturally would follow the apparatus necessary for the determination of the mineral constituents, but these, of course, would be such appliances as are found in every well-equipped chemical laboratory, no difference for what purpose it might be used. Next we would have a perfect illustration of all the appliances and methods used by the Association of Official Agricultural Chemists for the analysis of fertilizers and food products. To attempt a description of this class of apparatus, however, would only be to duplicate the reports of the Association of Official Agricultural Chemists, and hence reference is made to these reports to indicate the scope of the exhibition which should be made.

Especial attention should be paid to those branches of agricultural research of a chemical nature which are connected with great industries. For this purpose, a full illustration of the appliances used in the analysis of sugars and starches should be one of the prominent features of the exhibition; all the various kinds of polariscopes should be illustrated by the best instruments which the makers can produce, and, inasmuch as the operation of this class of instruments is easy, continued analyses of saccharine and carbohydrate products should be made a prominent feature of the work of the laboratory. A similar exhibit of apparatus used in the examination of other food products would naturally range themselves in line with the above.

Special attention, in this connection, should be given to methods of control which are used in the examination of sweet wines by the Bureau of Internal Revenue,

operations which, though not strictly belonging to agriculture, are particularly connected with agricultural products. The various methods of determining the alcohol should be illustrated, and all the chemical operations employed by the Bureau of Internal Revenue as applied to agricultural products should form a part of the exhibit.

In regard to the experts in charge, they should be selected according to the character of the work. There should be one person who should have charge especially of the physical examination of soils, another of the chemical examination, a third in charge of the examination of fertilizers, a fourth in charge of the part of the exhibit connected with the analysis of sugars, a fifth that of sweet wines; in short, it would seem that some six or eight experts, well skilled in the chemical branches which they are called upon to supervise, should be provided for the purpose of conducting the investigations to which allusion has been made. A mere collection of different kinds of apparatus inclosed in glass cases, in our opinion, would be of very little educational value. These appliances must be explained and illustrated by actual work.

Attention is particularly called to the fact that the aspect of the public toward such work has greatly changed in the last few years. Especially is this true of our agricultural population. The establishment of agricultural colleges and experiment stations has brought intimately to the notice of our prominent farmers the value of scientific research of the kind indicated. The patrons of the exhibit will be no idle on-lookers attending from curiosity; they will be made up largely of intelligent agronomists, who will bring with them the experience of actual conduct on their farms as the result of the scientific agricultural work of the various States. They will look with particular interest upon a practical illustration of the processes upon which they depend, for the purpose of securing a proper understanding of the nature of the material which they use for fertilizers, and the character of the products which are grown upon their farms. The above is the merest outline of what we consider should be the nature of the exhibition, but it is sufficient to show the general purpose we have in view, and the general character of the material which should be collected.

In the second place, we have to consider the means by which this exhibition can be maintained. Being of a national character we, of course, look first of all to Congress for the money necessary to maintain an exhibition of this description. When we consider the liberality of Congress in having endowed the vast system of agricultural colleges of the country with a fund which is as munificent as it is beneficial, and, in addition, when we consider the fact of the princely endowment of the agricultural experiment stations by our national Congress, we can not help feeling that an appeal to the same source would result in securing the necessary funds for the establishment of such an exhibit. There is already established, in connection with the Columbian Exposition, a department of agriculture, and an exhibit of chemical industries and appliances connected with agriculture would naturally fall under this department. We, therefore, should seek to secure a special appropriation, in connection with the department of agriculture of the Columbian Exposition, for the purpose of establishing a laboratory, such as has been described. This laboratory should be typical in every respect, not only for its educational value to the farmers themselves, but for the purpose of helping the various State agricultural colleges and experiment stations, many of which are now building laboratories for agricultural research. We should, therefore, ask for a sum which would be entirely sufficient to thoroughly equip a laboratory of this kind and to pay for its maintenance. Without going into the details of this equipment, it is sufficient to say that we should have at least $50,000 for this purpose. With such a sum it is believed that an exhibit could be made which would not only be a credit to American agriculture, but would show to the world at large the rapid progress which we are making in the scientific investigation of all subjects connected with our agricultural interests.

In view of the facts which have thus been generally outlined and submitted, we beg to make the following suggestions:

That the Association of Official Agricultural Chemists earnestly recommends the establishment, in connection with the Columbian Exposition of 1893, of a fully equipped and manned chemical laboratory for agricultural research.

That this body, representing as it does the agricultural interests of all the agricultural colleges and experiment stations of the United States, respectfully recommends to Congress the grant of an appropriation of $50,000 for the purpose of establishing, in connection with the department of agriculture of the Columbian Exposition, a laboratory for agricultural chemical research.

We further recommend that a committee be appointed for the purpose of presenting this matter to the Fifty-second Congress.

All of which is respectfully submitted.

H. W. WILEY.
NORMAN ROBINSON.

Mr. WILEY. Since the committee has adopted this report, the Secretary of Agriculture has given me authority to take official steps toward the establishment of a chemical laboratory on the part of the Department, provided the plan recommended in this report can not be accomplished.

Mr. FREAR. I inquire whether there has been any consultation with the committee of the Association of American Agricultural Colleges and Experiment Stations having an agricultural exhibit in view? I believe one thing proposed in connection with that is a chemical laboratory.

Mr. ROBINSON. I have had a consultation with Mr. Morrow, chairman of that committee, who assured me that he saw no possible conflict, and that he was cordially in favor of this movement.

On motion, the report of the committee was accepted and adopted.

Mr. FREAR presented the report of the committee on the proposed amendment to the constitution, as follows:

Mr. PRESIDENT: Your committee respectfully report the following proposed amendment to the constitution:

In section 2, last line, strike out the phrase, "have no vote in association," and insert "not be entitled to enter motions or vote."

WM. FREAR.
H. A. HUSTON.
H. W. WILEY.

On motion, the recommendations of the committee were adopted.

Mr. DE ROODE. The committee on the adoption of a system of atomic weights has decided to recommend the adoption of Clarke's latest atomic weights, but being undecided as to the figures, can not present them to the association until Prof. Clark can be consulted.

Mr. FREAR moved that the committee be continued, and be given power to draw up their report and insert it in the proceedings as a recommendation. This would not commit the association to its adoption. Carried.

Following is the report of the committee:

REPORT OF COMMITTEE ON ATOMIC WEIGHTS.[1]

The committee on the selection of a system of atomic weights to be used as official by the A. O. A. C. was continued, with power to act. Upon consultation with Prof. Clarke, we were advised to adopt the inclosed table. I sent the table to Dr. Jenkins and Mr. Voorhees, the other members of the committee, and I inclose their replies. The report of our committee is therefore as follows: "We advise the adoption of Prof. Clarke's table of atomic weights as revised for the United States Pharmacopœia December 6, 1890, the factor for the calculation of K_2O from K_2PtCl_6, however, being left unchanged."

Very respectfully,

RUDOLPH DE ROODE,
E. B. VOORHEES,
E. H. JENKINS,
Committee.

Mr. FREAR. I move that the secretary of the association be requested to convey to the authorities of the Columbian University thanks for the use of the building, and, for other courtesies extended, and also to the officials of the United States Department of Agriculture for the continuance of the many facilities afforded for the attainment of the objects of the association. Carried.

Mr. PATRICK. I came to this convention with a paper on dairy matters, but arrived too late to present it at the proper time. I ask whether the association will accept it for insertion in the proceedings. I do not care to take time to read it, as the hour for adjournment has arrived.

Mr. PAYNE. I move that it be inserted in the proceedings as a paper.[2] Carried.

The PRESIDENT. I desire to extend my thanks to the association for the hearty way in which my decisions have been accepted and my failings borne with.

The convention, on motion, adjourned to meet next year at the call of the executive committee.

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[1] For table of atomic weights recommended, see Methods of Analysis, p. 246.

[2] See section on Dairy Products, p. 26.

OFFICERS AND REPORTERS OF THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS OF THE UNITED STATES FOR 1890–'91.

PRESIDENT.

Mr. N. T. LUPTON, chemist, Alabama Agricultural Experiment Station.

VICE PRESIDENT.

Mr. S M. BABCOCK, chemist, Wisconsin Agricultural Experiment Station.

SECRETARY.

Mr. H. W. WILEY, chief chemist, U. S. Department of Agriculture.

EXECUTIVE COMMITTEE.

Mr. C. L. PENNY, of Newark, Del.
Mr. H. A. HUSTON, of La Fayette, Ind.

REPORTERS.

Phosphoric acid.—Mr. N. W. Lord, of Columbus, Ohio.
Nitrogen.—L. L. Van Slyke, of Geneva. N. Y.
Potash.—Mr. G. F. Payne, of Atlanta, Ga.
Soils and ash.—Mr. H. H. Harrington, of College Station, Tex.
Dairy products.—Mr. S. M. Babcock, of Madison, Wis.
Foods and feeding stuffs low in carbohydrates.—Mr. J. T. Anderson, of Auburn, Ala.
Foods and feeding stuffs high in carbohydrates.—Mr. W. H. Jordan, of Orono, Me.
Fermented liquors.—Mr. C. A. Crampton, of Washington, D. C.
Sugar.—Mr. B. B. Ross, of Baton Rouge, La.

CONSTITUTION OF THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS.

(1) This association shall be known as the Association of Official Agricultural Chemists of the United States. The objects shall be (1) to secure uniformity and accuracy of the methods, results, and modes of statements of analysis of fertilizers, soils, cattle foods,[1] dairy products, and other materials connected with agricultural industry; (2) to afford opportunity for the discussion of matters of interest to agricultural chemists.

(2) Analytical chemists connected with the United States Department of Agriculture, or with any State or national agricultural experiment station or agricultural college, or with any State or national institution or body charged with official control of the materials named in section 1, shall alone be eligible to membership, and one such representative for each of these institutions or boards, when properly accredited, shall be entitled to enter motions or vote in the association. Only such chemists as are connected with institutions exercising official fertilizer control shall vote on questions involving methods of analyzing fertilizers. Any person eligible to membership may become a member at any meeting of the association by presenting proper credentials and signing this constitution. All members of the association who lose their right to such membership by retiring from positions indicated as requisite for membership shall be entitled, to become honorary members and, to all privileges of membership save the right to hold office and vote. All analytical chemists and others interested in the objects of the association may attend its meetings and take part in its discussions, but shall not be entitled to enter motions or vote.

(3) The officers of the association shall consist of a president, a vice president, and a secretary who shall also officiate as treasurer; and these officers, together with two other members to be elected by the association, shall constitute the executive committee. When any officer ceases to be a member by reason of withdrawing from a department or board whose members are eligible to membership, his office shall be considered vacant, and a successor may be appointed by the executive committee, to continue in office till the annual meeting next following.

(4) There shall be appointed by the president, at the regular annual meeting, a reporter for each of the subjects to be considered by the association.

It shall be the duty of these reporters to prepare and distribute samples and standard reagents to members of the association and others desiring the same; to furnish blanks for tabulating analyses, and to present at the annual meeting the results of work done, discussion thereof, and recommendations of methods to be followed.

(5) The special duties of the officers of the association shall be further defined, when necessary, by the executive committee.

(6) The annual meeting of this association shall be held at such place as shall be decided by the association, and at such time as shall be decided by the executive committee, and announced at least three months before the time of meeting.

[1] By a vote of the association, August 30, 1890, the subject of cattle foods was divided into two heads, namely, foods and feeding stuffs It was understood by the latter of these divisions to include all fodders and feeding materials poor in starch and rich in fiber.

(7) Special meetings shall be called by the executive committee when in its judgment it shall be necessary, or on the written request of five members; and at any meeting, regular or special, seven enrolled members entitled to vote shall constitute a quorum for the transaction of business.

(8) The executive committee shall confer with the official boards represented, with reference to the payment of expenses connected with the meetings and publication of the proceedings of the association.

(9) All proposed alterations or amendments to this constitution shall be referred to a select committee of three, at a regular meeting, and after report from such committee, may be adopted by a vote of two-thirds of the members present entitled to vote.

OFFICIAL METHODS OF ANALYSIS ADOPTED BY THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS AT ITS MEETING AUGUST 13, 14, 15, 1891.[1]

METHODS FOR DETERMINING PHOSPHORIC ACID AND MOISTURE.

(1) *Preparation of sample.*—The sample should be well intermixed and properly prepared, and passed through a sieve having circular perforations one twenty-fifth of an inch in diameter, so that separate portions shall accurately represent the substance under examination, without loss or gain of moisture.

(2) *Determination of moisture.*—(*a*) In potash salts, nitrate of soda, and sulphate of ammonia, heat 1 to 5 grs. at 130° C. till the weight is constant, and reckon water from the loss. (*b*) In all other fertilizers heat 2 grs., or if the sample be too coarse to secure uniform lots of 2 grs. each, 5 grs. for five hours at 100° C. in a steam bath.

(3) *Water-soluble phosphoric acid.*—Weigh 2 grs.; transfer to a 9 cc. No. 589 Schleicher and Schüll filter, wash with water, allowing all the water to pass through each time before adding more, until the filtrate measures not less than 250 cc. Mix the washings. Take an aliquot portion (corresponding to 0.5 gr.) and determine phosphoric acid, as under total phosporic acid.

(4) *Citrate-insoluble phosphoric acid.*—Wash the residue of the treatment with water into a 200 cc. flask with 100 cc. of strictly neutral ammonium citrate solution of 1.09 density, prepared as hereafter directed. Cork the flask securely and place it in a water bath, the water of which stands at 65° C. (The water bath should be of such a size that the introduction of the cold flask or flasks shall not cause a reduction of the temperature of the bath of more than 2° C.) Raising the temperature as rapidly as practicable to 65° C., which is subsequently maintained, digest for thirty minutes from the instant of insertion, shaking every five minutes, filter the warm solution quickly (best with filter pump), and wash with water of about the temperature of 65°. In case of goods containing iron or alumina, the addition of a small amount of citrate solution to the wash water will give clear washings. Transfer the filter and its contents to a capsule, ignite until the organic matter is destroyed, treat with 10 to 15 cc. of concentrated hydrochloric acid, digest over a low flame until the phosphate is dissolved, dilute to 200 cc.; or treat the filter and its contents according to methods 1, 2, or 3 under total phosphoric acid; mix, pass through a dry filter, take an aliquot portion, and determine phosphoric acid as under total.

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[1] NOTE BY EDITOR.—It was voted by the association that each reporter should furnish the editor with a complete and corrected copy of the methods of analysis to be used on the particular subject of which he had charge. This request was complied with by Mr. H. A. Huston, reporter on phosphoric acid (with exception of the directions for washing water-soluble phosphoric acid on the filter and not to transfer to the beaker), Mr. W. C. Stubbs, reporter on sugar, Mr. R. C. Kedzie, reporter on soils, and by Messrs. Knorr and Woods, reporters on cattle foods. Mr. Battle, reporter on potash, furnished a list of the changes for incorporation in the method. In the other substances the editor had to be guided by the minutes of the association in regard to the changes adopted.

It is desirable that a clause be inserted in the constitution requiring each reporter to furnish, ready for the printer, a complete copy of the methods of analysis adopted at each meeting of the association.

In case a determination of citrate-insoluble phosphoric acid is required in non-acidulated goods, it is to be made by treating 2 grs. of the phosphatic material, without previous washing with water, precisely in the way above described, except that in case the substance contains much animal matter (bone, fish, etc.), the residue insoluble in ammonium citrate is to be treated by one of the processes described below under (5), 1, 2, or 3.

(5) *Total phosphoric acid.*—In case of ignition the ignited material is to be dissolved in hydrochloric acid. Weigh 2 grs. and treat by one of the following methods: (1) Evaporate with 5 cc. of magnesium nitrate, ignite, and dissolve in hydrochloric acid. (2) Boil with 20–30 cc. of strong sulphuric acid, adding 2–4 grs. of sodium or potassium nitrate at the beginning of the digestion, and a small quantity after the solution has become nearly colorless, or adding the nitrate in small portions from time to time. A Kjeldahl flask graduated to 250 cc. is recommended. After the solution is colorless add 150 cc. of water and boil for a few minutes, cool, and make up to volume. (3) Treat with sulphuric acid and mercury or mercuric oxide, as in the Kjeldahl method, using a flask graduated to 250 cc. When the digestion is complete add 150 cc. of water, boil for a few minutes, cool, and make up to volume. These methods are to be used in case of fertilizers containing cotton seed or much organic matter. (4) Solution in 30 cc. of concentrated nitric acid with a small quantity of hydrochloric acid. (5) Add 30 cc. of concentrated hydrochloric acid, heat, and add cautiously in small quantities at a time about 0.5 gr. of finely pulverized potassium chlorate. (6) Solution in 15–30 cc. of strong hydrochloric acid and 5–10 cc. of nitric acid. This method is recommended for fertilizers containing much phosphate of iron or alumina. Boil until all phosphates are dissolved and all organic matter is destroyed; cool and dilute to 200 or 250 cc.; mix and pass through a dry filter; take an aliquot part of the filtrate corresponding to 0.25 gr., 0.50 gr., or 1 gr.; neutralize with ammonia, and clear with a few drops of nitric acid. (In case hydrochloric or sulphuric acid has been used as a solvent, add about 15 grs. of dry ammonium nitrate or its equivalent.)

To the hot solutions for every decigram of P_2O_5 that is present, add 50 cc. of molybdic solution. Digest at about 65° C. for one hour, filter and wash with water or ammonium nitrate solution. (Test the filtrate by renewed digestion and addition of more molybdic solution.) Dissolve the precipitate on the filter with ammonia and hot water, and wash into a beaker to a bulk of not more than 100 cc. Nearly neutralize with hydrochloric acid, cool, and add magnesia mixture from a burette; add slowly (one drop per second), stirring vigorously. After fifteen minutes add 30 cc. of ammonia solution of density 0.95. Let stand several hours (two hours is usually enough). Filter, wash with dilute ammonia, ignite intensely for ten minutes, and weigh.

(6) *Citrate-soluble phosphoric acid.*—The sum of the water-soluble and citrate-insoluble subtracted from the total gives the citrate-soluble.

PREPARATION OF REAGENTS.

(1) *To prepare ammonium citrate solution.*—(*a*) Mix 370 grs. of commercial citric acid with 1,500 cc. of water; nearly neutralize with commercial ammonia; cool; add ammonia until exactly neutral (testing with saturated alcoholic solution of coraline) and bring to volume of 2 liters. Test the specific gravity, which should be 1.09 at 20° C., before using.

(*b*) Alternate method. To 370 grs. of commercial citric acid add commercial ammonia, sp. gr. 0.96, until nearly neutral; reduce the specific gravity to nearly 1.09 and make exactly neutral, testing as follows: Prepare a solution of fused calcium chloride, 200 grs. to the liter, and add four volumes of strong alcohol. Make the mixture exactly neutral, using a small amount of freshly prepared coralline solution as a preliminary indicator, and test finally by withdrawing a portion, diluting with an equal volume of water and testing with cochineal solution. 50 cc. of this solution will precipitate the citric acid from 10 cc. of the citrate solution. To 10 cc. of

the nearly neutral citrate solution add 50 cc. of the alcoholic chloride of calcium solution, stir well, filter at once through a folded filter, dilute with an equal volume of water, and test the reaction with neutral solution of cochineal.

(2) *To prepare molybdic solution.*—Dissolve 100 grs. of molybdic acid in 400 grs., or 417 cc., of ammonia sp. gr. 0.96, and pour the solution thus obtained into 1,500 grs., or 1,250 cc. of nitric acid sp gr. 1.20. Keep the mixture in a warm place for several days, or until a portion heated to 40° C. deposits no yellow precipitate of ammonium phospho-molybdate. Decant the solution from any sediment, and preserve in glass-stoppered vessels.

(3) *To prepare ammonium nitrate solution.*—Dissolve 200 grs. of commercial ammonium nitrate in water, and bring to a volume of 2 liters.

(4) *To prepare magnesia mixture.*—Dissolve 22 grs. of recently ignited calcined magnesia in dilute hydrochloric acid, avoiding an excess of the latter. Add a little calcined magnesia in excess, and boil a few minutes to precipitate iron, alumina, and phosphoric acid; filter; add 280 grs. of ammonium chloride, 700 cc. of ammonia of sp. gr. 0.96, and water enough to make a volume of 2 liters. Instead of the solution of 22 grs. of calcined magnesia 110 grs. of crystallized magnesium chloride ($MgCl_2 + 6H_2O$) may be used.

(5) *Dilute ammonia for washing.*—One volume of ammonia sp. gr. 0.96 mixed with three volumes of water, or usually one volume of concentrated ammonia with six volumes of water.

(6) *Nitrate of magnesia.*—Dissolve 320 grs. of calcined magnesia in nitric acid, avoiding an excess of the latter; then add a little calcined magnesia in excess; boil; filter from the excess of magnesia, ferric oxide, etc., and bring to volume of 2 liters.

METHODS FOR THE DETERMINATION OF NITROGEN.

THE ABSOLUTE OR CUPRIC OXIDE METHOD.

[Applicable to all nitrogen determinations.]

The apparatus and reagents needed are as follows:

APPARATUS.

Combustion tube of best hard Bohemian glass, about 26 inches long and one-half inch internal diameter.

Azotometer of at least 100 cc. capacity, accurately calibrated.

Sprengel mercury air-pump.

Small paper scoop, easily made from stiff writing paper.

REAGENTS.

Cupric oxide (coarse).—Wire form; to be ignited and cooled before using.

Fine cupric oxide.—Prepared by pounding ordinary cupric oxide in a mortar.

Metallic copper.—Granulated copper, or fine copper gauze, reduced and cooled in a current of hydrogen.

Sodium bicarbonate.—Free from organic matter.

Caustic potash solution.—Dissolve commercial stick potash in less than its weight of water, so that crystals are deposited on cooling. When absorption of carbonic acid ceases to be prompt, the solution must be discarded.

LOADING THE TUBE.

Of ordinary commercial fertilizers take 1 to 2 grs. for analysis. In the case of highly nitrogenized substances the amount to be taken must be regulated by the amount of nitrogen estimated to be present. Fill the tube as follows: (1) About 2 inches of coarse cupric oxide. (2) Place on the small paper scoop enough of the fine cupric

oxide to fill, after having been mixed with the substance to be analyzed, about 4 inches of the tube; pour on this the substance, rinsing the watch glass with a little of the fine oxide, and mix thoroughly with a spatula; pour into the tube, rinsing the scoop with a little fine oxide. (3) About 12 inches of coarse cupric oxide. (4) About 3 inches of metallic copper. (5) About 2½ inches of coarse cupric oxide (anterior layer). (6) A small plug of asbestos. (7) 0.8 to 1 gr. of sodium bicarbonate. (8) A large, loose plug of asbestos; place the tube in the furnace, leaving about 1 inch of it projecting; connect with the pump by a rubber stopper smeared with glycerin, taking care to make the connection perfectly tight.

OPERATION.

Exhaust the air from the tube by means of the pump. When a vacuum has been obtained allow the flow of mercury to continue; light the gas under that part of the tube containing the metallic copper, the anterior layer of cupric oxide (see 5th above), and the bicarbonate of soda. As soon as the vacuum is destroyed and the apparatus filled with carbonic acid gas, shut off the flow of mercury and at once introduce the delivery tube of the pump into the receiving arm of the azotometer just below the surface of the mercury seal of the azotometer, so that the escaping bubbles will pass into the air and not into the azotometer, thus avoiding the useless saturation of the caustic-potash solution.

When the flow of carbonic acid has very nearly or completely ceased, pass the delivery tube down into the receiving arm, so that the bubbles will escape into the azotometer. Light the jets under the 12-inch layer of oxide, heat gently for a few moments to drive out any moisture that may be present, and bring to red heat. Heat gradually the mixture of substance and oxide, lighting one jet at a time. Avoid a too rapid evolution of bubbles, which should be allowed to escape at the rate of about one per second or a little faster.

When the jets under the mixture have all been turned on, light the jets under the layer of oxide at the end of the tube When the evolution of gas has ceased, turn out all the lights except those under the metallic copper and anterior layer of oxide, and allow to cool for a few moments. Exhaust with the pump and remove the azotometer before the flow of mercury is stopped. Break the connection of the tube with the pump, stop the flow of mercury, and extinguish the lights. Allow the azotometer to stand for at least an hour, or cool with a stream of water until a permanent volume and a temperature have been reached.

Adjust accurately the level of the KOH solution in bulb to that in the azotometer; note the volume of gas, temperature, and height of barometer; make calculations as usual. The labor of calculation may be much diminished by the use of the tables prepared by Messrs. Battle and Dancy, of the North Carolina Experiment Station (Raleigh, N. C.).

The above details are, with some modifications, those given in the report of the Connecticut Station for 1878 (p. 124), which may be consulted for details of apparatus, should such details be desired.

THE KJELDAHL METHOD.

[Not applicable in presence of nitrates.]

APPARATUS AND REAGENTS.

(1) Standard hydrochloric acid the absolute strength of which has been determined: (*a*) By precipitating with silver nitrate and weighing the silver chloride as described in Crook's Select Methods, page 571; (*b*) by potassium tetra-oxalate which has been purified by recrystallizing two or three times; (*c*) by determining the amount neutralized in the distillation of a weighed quantity of pure ammonium chloride boiled with an excess of sodium hydrate. Half normal acid, hydrochloric acid *i. e.*, containing 18.23 grs. to the liter, is recommended. Cochineal is used as the indicator in standardizing the acid.

(2) Standard ammonia the strength of which, relative to the acid, has been accurately determined. One-tenth normal ammonia, *i. e.*, containing 0.7 gr. to the liter, is recommended for accurate work.

(3) "C. P." sulphuric acid, sp. gr. 1.83, free from nitrates and also from ammonic sulphate, which is sometimes added in the process of manufacture to destroy oxides of nitrogen.

(4) Metallic mercury or mercuric oxide, HgO, prepared in the wet way. That prepared from mercuric nitrate can not be safely used.

(5) Potassium permanganate tolerably finely pulverized.

(6) Granulated zinc, pumice stone, or 0.5 gr. of zinc dust are to be added to the contents of the flasks in distillation, when found necessary, in order to prevent bumping.

(7) A solution of 40 grs. of commercial potassium sulphide in one liter of water.

(8) A saturated solution of sodium hydrate free from nitrates, which are sometimes added in the process of manufacture to destroy organic matter and improve the color of the product.

(9) Solution of cochineal prepared according to Fresenius's Quantitative Analysis, second American edition, page 679.

(10) Kjeldahl digestion flasks of hard, moderately thick, well-annealed glass. These flasks are about 9 inches long, with a round, pear shaped bottom, having a maximum diameter of 2½ inches, and tapering out gradually in a long neck, which is three-fourths of an inch in diameter at the narrowest part, and flared a little at the edge. The total capacity is 225 to 250 cc.

(11) Distillation flasks of ordinary shape, of 550 cc. capacity, or preferably flasks of the same capacity, of pear-shaped bottom, of well-annealed glass, for both digestion and distillation, fitted with a rubber stopper and a bulb tube above to prevent the possibility of sodium hydrate being carried over mechanically during distillation. The bulbs are about 1½ inches in diameter, the tubes being of the same diameter as the condenser and cut off obliquely at the lower end. This is adjusted to the tube of the condenser by a rubber tube.

(12) *A condenser.*—Several forms have been described, no one of which is equally convenient for all laboratories. The essential thing is that the tube which carries the steam to be condensed shall be of block tin. The upper ends of the tin tubes should be bent, so that the glass connections may have a slope toward the distilling flasks. All kinds of glass are decomposed by steam and ammonia vapor, and will give up alkali enough to impair accuracy. (See Kreussler and Henzold, Ber. Deut. Chem. Ges., XVII, 34.) The condenser in use in the laboratory of the Connecticut Experiment Station, devised by Prof. Johnson, consists of a copper tank, supported by a wooden frame, so that its bottom is 11 inches above the work-bench on which it stands. This tank is 16 inches high, 32 inches long, and 3 inches wide from front to back, widening above to 6 inches. It is provided with a water-supply tube, which goes to the bottom, and a larger overflow pipe above. The block tin condensing tubes, the external diameter of which is three-eighths of an inch, 7 in number, enter the tank through holes in the front side of it near the top, above the level of the overflow, and pass down perpendicularly through the tank, and out through rubber stoppers tightly fitted into holes in the bottom. They project about 1½ inches below the bottom of the tank, and are connected by short rubber tubes with glass bulb tubes of the usual shape, which dip into precipitating beakers or Erlenmeyer flasks of about 300 cc. capacity. The titration can be made directly in them. The distillation flasks are supported on a sheet-iron shelf, attached to the wooden frame that supports the tanks, in front of the latter. Where each flask is to stand, a circular hole is cut, with three projecting lips, for supporting the wire gauze or asbestos under the flask, and three other lips, which hold the flask in place and prevent its moving laterally while distillation is going on. Below this sheet-iron shelf is a metal tube carrying seven Bunsen burners, each with a stopcock like those of a gas combustion furnace. These burners are of a larger diameter at the top, to avoid smoking when covered with fine gauze to prevent the flame from striking back.

(13) The stand for holding the digestion flasks consists of a pan of sheet iron 29 inches long by 8 inches wide, on the front of which is fastened a shelf of sheet-iron as long as the pan, 5 inches wide and 4 inches high. In this are cut six holes 1⅝ inches in diameter. At the back of the pan is a stout wire running lengthwise of the stand, 8 inches high, with a bend or depression opposite each hole in the shelf. The digestion flask rests with its lower part over a hole in the shelf and its neck in one of the depressions in the wire frame, which holds it securely in position. Heat is supplied by low Bunsen burners below the shelf. With a little care the naked flame can be applied directly to the flask without danger.

THE DETERMINATION.

(1) *The digestion.*—0.7 gr. to 2.8 gr. of the substance to be analyzed, according to its proportion of nitrogen, is brought into a digestion flask with approximately 0.7 gr. of mercuric oxide, or its equivalent in metallic mercury, and 20 cc. of sulphuric acid. The flask is placed on the frame above described in an inclined position, and heated below the boiling point of the acid for from five to fifteen minutes, or until frothing has ceased. If the mixture froths badly, a small piece of paraffine may be added to prevent it. The heat is then raised until the acid boils briskly. No further attention is required till the contents of the flask have become a clear liquid, which is colorless, or at least has only a very pale straw color. The flask is then removed from the frame, held upright, and while still hot, potassium permanganate is dropped in carefully and in small quantities at a time till, after shaking, the liquid remains of a green or purple color.

(2) *The distillation.*—After cooling, the contents of the flask are transferred to the distilling flask with about 200 cc. of water, with a few pieces of granulated zinc, pumice stone, or 0.5 gr. of zinc dust when found necessary to keep the contents of the flask from bumping, and 25 cc. of potassium sulphide solution are added, shaking the flask to mix its contents. Next add 50 cc. of the soda solution, or sufficient to make the reaction strongly alkaline, pouring it down the side of the flask so that it does not mix at once with the acid solution. Connect the flask with the condenser, mix the contents by shaking, and distill until all ammonia has passed over into the standard acid. The first 150 cc. of the distillate will generally contain all of the ammonia. This operation usually requires from forty minutes to one hour and a half. The distillate is then titrated with standard ammonia.

The use of mercuric oxide in this operation greatly shortens the time necessary for digestion, which is rarely over an hour and a half in case of substances most difficult to oxidize, and is more commonly less than an hour. In most cases the use of potassium permanganate is quite unnecessary, but it is believed that in exceptional cases it is required for complete oxidation, and in view of the uncertainty it is always used. The Potassium sulphide removes all the mercury from the solution, and so prevents the formation of mercurammonium compounds which are not completely decomposed by soda solution. The addition of zinc gives rise to an evolution of hydrogen, and prevents violent bumping. Previous to use the reagents should be tested by a blank experiment with sugar, which will partially reduce any nitrates that are present, which might otherwise escape notice.

KJELDAHL METHOD MODIFIED TO INCLUDE THE NITROGEN OF NITRATES.[1]

[Applicable to all fertilizers containing nitrates.]

Besides the reagents and apparatus given under the Kjeldahl method there will be needed—

(1) Zinc dust. This should be an impalpable powder; granulated zinc or zinc filings will not answer.

(2) Zinc sulphide.

(3) Commercial salicylic acid.

[1] Described by Prof. M. A. Scovell.

THE DETERMINATION.

Bring from 0.7 to 1.4 grs. of the substance to be analyzed into a Kjeldahl digesting flask, add to this 30 cc. of sulphuric acid containing 2 grs. of salicylic acid, and shake thoroughly; then add *gradually* 2 grs. of zinc dust, shaking the contents of the flask at the same time, or add to the substance 30 cc. of sulphuric acid containing 1 gr. of salicylic acid; then add direct 2 grs. of zinc sulphide or 5 grs. of cystallized sodium hyposulphite. Finally place the flask on the stand for holding the digestion flasks, where it is heated over a low flame until all danger from frothing has passed. The heat is then raised until the acid boils briskly, and the boiling continued until white fumes no longer pour out of the flask. This requires about five or ten minutes. Add now approximately 0.7 gr. of mercuric oxide, or its equivalent in metallic mercury, and continue the boiling until the liquid in the flask is colorless, or nearly so. (In case the contents of the flask are likely to become solid before this point is reached, add 10 cc. more of sulphuric acid.) Complete the oxidation with a little permanganate of potash in the usual way, and proceed with the distillation as described in the Kjeldahl method. The reagents should be tested by blank experiments.

THE RUFFLE METHOD.

APPARATUS AND REAGENTS.

(1) Standard solutions and indicator the same as for the Kjeldahl method.

(2) A mixture of equal parts by weight of fine slaked lime and finely powdered sodium hyposulphite dried at 100° C.

(3) A mixture of equal parts by weight of finely powdered granulated sugar and flowers of sulphur.

(4) Granulated soda-lime, as described under the soda-lime method.

(5) Combustion tubes of hard Bohemian glass, 20 inches long and one-half inch in diameter.

(6) Bulbed "U" tubes or Will's bulbs, as described under the soda-lime method.

PREPARATION.

(1) Clean the U tube and introduce 10 cc. of standard acid.

(2) Fit the cork and glass connecting tube. Fill the tube as follows: (1) A loosely fitting plug of asbestos, previously ignited, and then 1 to 1½ inches of the hyposulphite mixture. (2) The weighed portion of the substance to be analyzed is intimately mixed with from 5 to 10 grs. of the sugar and sulphur mixture. (3) Pour on a piece of glazed paper or in a porcelain mortar a sufficient quantity of hyposulphite mixture to fill about 10 inches of tube; then add the substance to be analyzed, as previously prepared; mix carefully and pour into the tube; shake down the contents of the tube; rinse off the paper or mortar with a small quantity of the hyposulphite mixture and pour into the tube; then fill up with soda-lime to within 2 inches of the end of the tube. (4) Place another plug of ignited asbestos at the end of the tube and close with a cork. (5) Hold the tube in a horizontal position and tap on the table until there is a gas channel all along the top of the tube. Make connection with the U tube containing the acid; aspirate, and see that the apparatus is tight.

The combustion.—Place the prepared combustion tube in the furnace, letting the open end project a little so as not to burn the cork. Commence by heating the soda-lime portion until it is brought to a full red heat. Then turn on slowly jet after jet toward the outer end of the tube, so that the bubbles come off two or three a second. When the whole tube is red hot and the evolution of the gas has ceased, and the liquid in the U tube begins to recede toward the furnace, attach the aspirator to the other limb of the U tube, break off the end of the tube, and draw a current of air through for a few minutes. Detach the U tube and wash the contents into a beaker or porcelain basin, add a few drops of the cochineal solution, and titrate.

THE SODA-LIME METHOD.

[Not applicable in presence of nitrates.]

APPARATUS AND REAGENTS.

(1) Standard solutions and indicator the same as for the Kjeldahl method.

(2) Granulated soda-lime, fine enough to pass a 10-mesh sieve, and thoroughly dry.

(3) Fine soda-lime, fine enough to pass a 20-mesh sieve, also thoroughly dry.

To prepare soda-lime of the required fineness, the coarse granulated article of the trade may be ground until it will pass through a sieve of about ten-hundredths inch mesh. It is then sifted on a sieve of about twenty-hundredths inch mesh to separate the fine from the coarse. The two portions are then thoroughly dried in the air bath, or by heating in a porcelain dish or iron pan over a lamp, stirring constantly.

Excellent soda-lime may be easily and cheaply prepared according to the directions of Prof. Atwater (Am. Chem. Journal, vol. 9, p. 312) by slaking 2½ parts of quicklime with a strong solution of 1 part of commercial caustic soda (such soda as is used in the Kjeldahl process), care being taken that there is enough water in the solution to slake the lime. The mixture is then dried and heated in an iron pot to incipient fusion, and when cold, ground and sifted as above.

Instead of soda-lime, Johnson's mixture of carbonate of soda and lime, or slaked lime may be used.

Slaked lime may be granulated by mixing it with a little water to form a thick mass, which is dried in the water oven until hard and brittle. It is then ground and sifted as above. Slaked lime is much easier to work with than soda-lime, and gives excellent results, though it is probable that more of it should be used in proportion to the substance to be analyzed than is the case with soda-lime.

(4) Asbestos which has been ignited and kept in a glass-stoppered bottle.

(5) Combustion tubes about 40 cm. long and of 12 mm. internal diameter, drawn out to a point and closed at one end.

(6) Large-bulbed "U" tubes with glass stopcock, or Will's tubes with four bulbs.

THE DETERMINATION.

The substance to be analyzed should be powdered fine enough to pass through a sieve of 1 mm. mesh; 0.7 to 1.4 grs., according to the amount of nitrogen present, is taken for the determination. Into the closed end of the combustion tube put a small loose plug of asbestos, and upon it about 4 cm. of fine soda-lime. In a porcelain dish or mortar mix the substance to be analyzed thoroughly, but quickly, with enough fine soda-lime to fill about 16 cm. of the tube, or about 40 times as much soda-lime as substance, and put the mixture into the combustion tube as quickly as possible by means of a wide-necked funnel, rinsing out the dish and funnel with a little more fine soda-lime, which is to be put in on top of the mixture. Fill the rest of the tube to about 5 cm. of the end with granulated soda-lime, making it as compact as possible by tapping the tube gently while held in a nearly upright position during the filling. The layer of granulated soda-lime should be not less than 12 cm. long. Lastly, put in a plug of asbestos about 2 cm. long, pressed rather tightly, and wipe out the end of the tube to free it from adhering soda-lime.

Connect the tube by means of a well-fitting rubber stopper or cork with the "U" tube or Will's bulbs, containing 10 cc. of standard acid, and adjust it in the combination furnace so that the end projects about 4 cm. from the furnace, supporting the "U" tube or Will's bulb suitably. Heat the portion of the tube containing the granulated soda-lime to a moderate redness, and when this is attained extend the heat gradually through the portion containing the substance so as to keep up a moderate and regular flow of gases through the bulbs, maintaining the heat of the first part until the whole tube is heated uniformly to the same degree. Keep up the heat until gases have ceased bubbling through the acid in the bulbs, and the mixture of substance and soda-

lime has become white, or nearly so, which shows that the combustion is finished. The combustion should occupy about three-quarters of an hour, or not more than one hour. Remove the heat, and when the tube has cooled below redness break off the closed tip and aspirate air slowly through the apparatus for two or three minutes to bring all the ammonia into the acid. Disconnect, wash the acid into a beaker or flask, and titrate with the standard alkali.

During the combustion the end of the tube projecting from the furnace must be kept heated sufficiently to prevent the condensation of moisture, yet not enough to char the stopper. The heat may be regulated by a shield of tin slipped over the projecting end of the combustion tube.

It is found very advantageous to attach a Bunsen valve to the exit tube, allowing the evolved gases to pass out freely, but preventing a violent "sucking back" in case of a sudden condensation of steam in the bulbs.

METHOD OF DETERMINING POTASH.

METHOD OF LINDO-GLADDING.

(1) *In superphosphates.*—Boil 10 grs. with 300 cc. of water for thirty minutes. To the hot solution[1] add ammonia in slight excess, and then a little oxalate of ammonia, thus precipitating all phosphate and sulphate of lime, oxide of iron and alumina, etc.; make up to 500 cc., mix thoroughly, and filter through a dry filter; take 50 cc., corresponding to 1 gr., evaporate nearly to dryness, add 1 cc. of dilute H_2SO_4 (1 to 1), evaporate to dryness and ignite to whiteness. As all the potash is in form of sulphate, no loss need be apprehended by volatilization of potash, and a full red heat must be maintained until the residue is perfectly white. This residue is dissolved in hot water, plus a few drops of HCl; 5 cc. of a solution of pure NaCl (containing 20 grs. NaCl to the liter), or its equivalent of dry salt, and an excess of platinum solution (4 cc.) are now added. This solution is then evaporated to a thick paste in a small dish, and 80 per cent alcohol added In evaporating, special precaution should be taken to prevent absorption of ammonia. The precipitate is washed thoroughly with alcohol by decantation and on the filter, as usual. The washing should be continued even after the filtrate is colorless. 10 cc. of the NH_4Cl solution, prepared as hereafter directed, are now run through the filter, or the washing may be performed in the dish. This 10 cc. will contain the bulk of the impurities, and is thrown away. Fresh portions of 10 cc. of the NH_4Cl are now run through the filter several times (five or six). The filter is then washed thoroughly with pure alcohol, dried, and weighed as usual. Care should be taken that the precipitate is perfectly soluble in water. The platinum solution used contains 1 gr of metallic platinum in every 10 cc. To prepare the washing solution of NH_4Cl, place in a bottle 500 cc. of H_2O, 100 grs. of NH_4Cl; shake till dissolved. Now pulverize 5 or 10 grs. of K_2PtCl_6, put in the bottle, and shake at intervals for six or eight hours; let settle over night, then filter off the liquid into a second bottle. The first bottle is then ready for preparation of a fresh supply when needed.

(2) *Muriates of potash.*—In the analysis of these salts an aliquot portion, containing 0.5 gr., is evaporated with 10 cc. of platinum solution, and a few drops of HCl, and washed as before.

Amendment to section 3 of the Lindo-Gladding method for potash (proceedings of 1890, p. 210):

(3) *Sulphate of potash, kainite, etc.*—In the analysis of kainit, dissolve 10 gr. of the pulverized salt in 300 cc. of boiling water, add ammonia to slight excess, then a little oxalate of ammonia; make up to 500 cc., mix thoroughly, and filter on a dry filter; take 25 cc., corresponding to 0.5 gr., and proceed to remove the ammonia, as in

[1] The phrase "to the hot solution" was omitted from the method published in Bull. 28, but should be used in the description of the official methods. (See Bull. 28, pp. 74, 75).

the analysis of superphosphates; dissolve the residue in hot water, plus a few drops of HCl, add 0.25 gr. of NaCl and 15 cc. of platinum solution. In the analysis of high-grade sulphate and of double-manure salt, make up the solution as above, but omit the precipitation, evaporation, etc.; take an aliquot part equal to 0.5 gr., and add 0.25 gr. NaCl and 15 cc. of platinum solution. In these cases special care must be taken in the washing with alcohol to remove all the double chloride of platinum and sodium. The washing should be continued for some time after the filtrate is colorless. 25 cc. of the NH_4Cl solution are employed instead of 10 cc., and the 25 cc. poured through at least six times to remove all sulphates and chlorides. Wash finally with alcohol, dry, and weigh as usual.

(4) *Organic compounds.*—In case the potash is contained in organic compounds like tobacco stems, cotton-seed hulls, etc., weigh out 10 grs., saturate with strong sulphuric acid, and ignite in a muffle to destroy organic matter. Add a little strong HCl to moisten the mass, and warm slightly so as to loosen it in the dish. Proceed then as in (1).

ALTERNATE METHODS.

Prepare the fertilizer as for the Lindo-Gladding method; take 10 grs., boil for ten minutes with 200 cc. of water, and, after cooling and without filtering, make up to 1,000 cc. and filter through a dry filter. If the sample has 10 to 15 per cent of K_2O (kainit), take 50 cc. of the filtrate, if from 2 to 3 per cent of K_2O (ordinary potash fertilizers), take 100 cc. of the filtrate. In each case make the volume up to 150 cc., heat to 100° C., and add, drop by drop with constant stirring, slight excess of barium chloride; without filtering, in the same manner, add barium hydrate in slight excess. Heat, filter, and wash until the precipitate is free of chlorides. Add to the filtrate 1 cc. of strong ammonium hydrate, and then a saturated solution of ammonium carbonate, until the excess of barium is precipitated. Heat. Add now, in fine powder, 0.5 gr. pure of oxalic acid or 0.75 gr. of ammonium oxalate. Filter; wash free of chlorides; evaporate the filtrate to dryness in a platinum dish, and, holding the dish with crucible tongs, ignite carefully over the free flame below red heat until all volatile matter is driven off.

The residue is now digested with hot water, filtered through a small filter, and washed with successive small portions of water, until the filtrate amounts to 30 cc. or more. To this filtrate, after adding two drops of strong hydrochloric acid, is added, in a porcelain dish, 5 to 10 cc. of a solution of 10 grs. of platinic chloride in 100 cc. of water. The mixture is now evaporated on the water bath to a thick sirup, as above, treated with 80 per cent alcohol, washed by decantation, collected in a Gooch crucible or other form of filter, washed with strong alcohol, afterwards with 5 cc. of ether, dried for thirty minutes at 100° C, and weighed.

It is desirable, if there be an appearance of foreign matter in the double salt, that it should be washed, according to the previous method, with 10 cc. of the half concentrated solution of NH_4Cl, which has been saturated by shaking with K_2PtCl_6, as recommended by Gladding.

The use of the factors 0.3056 for converting K_2PtCl_6 to KCl, and 0.19308 for converting to K_2O, is continued.

THE OFFICIAL METHOD FOR THE ANALYSIS OF CATTLE FOODS FOR 1891–'92.

PREPARATION OF THE SAMPLE.

The substance is to be ground till all of it will pass through a sieve with circular holes 1 millimeter in diameter.

HYGROSCOPIC WATER.

Dry 2 to 3 grs. of the substance for four hours, heating fully to the temperature of boiling water, in a current of dry hydrogen, but without allowing the glass containing it to come in contact with the boiling water.

ASH.

Char 2 to 3 grs. of the substance, and burn to whiteness at the lowest possible red heat; this may be conveniently done in a Fletcher gas-muffle furnace, heated not by the large gas burner that is supplied with the furnace, but by a Fletcher solid-flame burner (No. 466).

If a white ash can not be obtained in this manner, exhaust the charred mass with water; collect the insoluble residue on a filter, burn, add this ash to the residue from the evaporation of the above aqueous extract, and heat the whole to low redness till the ash is white.

ETHER EXTRACT.

Extract 2 to 3 grs. of the substance dried as for the determination of the moisture, with specially prepared anhydrous and alcohol-free ether, sixteen hours. Dry the extract, by exposure to the full heat of boiling water, to constant weight.

Alternate method.—In determining hygroscopic water, as above, continue the drying until the loss of weight in 30 minutes is reduced to 1 milligram, extract the dried substance for sixteen hours as directed, dry again, and give loss of weight as ether extract.

The determination of fat by difference may thus be made to check the determination by weight.

To prepare the anhydrous alcohol-free ether required for estimation of fat, take Squibb's or any of the commercial brands of ether, wash with 2 or 3 successive portions of distilled water, add sticks of solid caustic soda or potash until most of the water has been abstracted from the ether, which is then drawn off into a stock bottle. Carefully cleaned metallic sodium, cut into small pieces, is now added until there is no further evolution of hydrogen gas.

The ether thus dehydrated must be kept over metallic sodium, and should be only lightly stoppered, in order to allow the accumulating hydrogen gas to escape; and it may be drawn off with a pipette as required. No sodium will go into solution if proper care is employed in removing the rind from the metal before adding to the ether.

CRUDE PROTEIN.

Determine nitrogen by the Kjeldahl method as directed for nitrogen in fertilizers, and multiply the result by 6.25 for the crude protein.

ALBUMINOID NITROGEN.

Stutzer's method.—Prepare cupric hydrate as follows: Dissolve 100 grs. of pure cupric sulphate in 5 liters of water, and add 25 cc. of glycerine; add dilute solution of sodium hydrate until the liquid is alkaline; filter; rub the precipitate up with water containing 5 cc. of glycerine per liter, and then wash by decantation or filtration until the washings are no longer alkaline. Rub the precipitate up again in a mortar with water containing 10 per cent of glycerine, thus preparing a uniform gelatinous mass that can be measured out with a pipette. Determine the quantity of cupric hydrate per cubic centimeter of this mixture. To 0.7 to 0.8 gr. of the substance add 100 cc. of water in a beaker, heat to boiling, or in the case of substances rich in starch heat on the water bath ten minutes, add a quantity of cupric hydrate mixture containing 0.5 to 0.6 gr. of the hydrate, stir thoroughly, filter when cold, wash with cold water, and put the filter and its contents into the concentrated sulphuric acid for the determination of nitrogen after Kjeldahl. Add sufficient potassium sulphide solution to completely precipitate all copper and mercury.

For the above filtration use Schleicher and Schüll's No. 589 paper, or Swedish paper, either of which contains so little nitrogen that it can be left out of account.

If the substance examined consist of seed of any kind, or residues of seeds, such as oil cake or anything else rich in alkaline phosphates, add a few cubic centimeters of a concentrated solution of alum just before adding the cupric hydrate, and mix well by stirring. This serves to decompose the alkaline phosphates. If this is not done cupric phosphate and free alkali may be formed, and the protein copper may be partially dissolved in the alkaline liquid.

CRUDE FIBER.

(*a*) *For feeding stuffs poor in starch.*—Extract 2 grs. of the substance with ordinary ether, at least nearly completely, or take the residue from the determination of the ether extract. To this residue, in a 500 cc. (Erlenmeyer) flask, add 200 cc. of boiling 1.25 per cent sulphuric acid; connect the flask with a return-flow condenser, the tube of which passes only a short distance beyond the rubber stopper into the flask. Boil at once, and continue the boiling for thirty minutes. A blast of air conducted into the flask may serve to reduce the frothing of the liquid.

Filter; wash thoroughly with boiling water till the washings are no longer acid; rinse the substance back into the same flask with 200 cc. of a boiling 1.25 per cent solution of sodium hydrate at least almost free from sodium carbonate; boil at once, and continue the boiling for thirty minutes in the same manner as directed above for the treatment with acid. Filter in a Gooch crucible, and wash with boiling water till the washings are neutral; dry at 110° C.; weigh; incinerate completely, and give the loss of weight for crude fiber.

The filter used for the first filtration may be linen, one of the forms of glass wool or asbestos filters recommended in the last report, or any other form that secures clear and reasonably rapid filtration. The solutions of sulphuric acid and sodium hydrate are to be made up of the specified strength accurately by titration, and not merely by the areometer.

(*b*) *For feeding stuffs rich in starch.*—Proceed as under (*a*) using 2.5 per cent acid and alkali in place of the solution of 1.25 per cent strength.

METHODS OF ANALYSIS OF DAIRY PRODUCTS.

BUTTER.

MICROSCOPIC EXAMINATION.

Place a small portion of the fresh sample taken from the inside of the mass, on a slide, add a drop of pure sweet oil, cover with gentle pressure, and examine with a one-half to one-eighth inch objective for crystals of lard, etc.

Examine the same specimen with polarized light and a selenite plate without the use of oil.

Pure fresh butter will neither show crystals nor a particolored field with selenite. Other fats melted and cooled, and mixed with butter, will usually present crystals and variegated colors with the selenite plate.

For further microscopic study dissolve 4 or 5 cc. of the fat in 15 cc. of ether in a test tube. Close the tube with a loose plug of cotton wool and allow to stand twelve to twenty-four hours at room temperature (20° C. to 25° C.). When crystals form at the bottom of the tube they are removed with a pipette, glass rod, or tube, placed on a slide, covered, and examined. The crystals formed by later deposits may be examined in a similar way.

Sampling.—If large quantities of butter are to be sampled a butter trier or sampler may be used. The portions thus drawn, about 500 grs, are to be perfectly melted in a closed vessel at as low a heat as possible, and when melted, the whole is to be shaken violently for some minutes till the mass is homogeneous. A portion is then poured into a vessel from which it is to be weighed out for analysis, and should nearly or quite fill it. This sample should be kept in a cold place till analyzed.

Determination of water.—1.5 to 2.5 grs. is dried to constant weight at the temperature of boiling water in a dish with flat bottom, having a surface of at least 20 cm^2.

The use of clean dry sand or asbestos with the butter is admissible, and is necessary if a dish with round bottom is employed.

Determination of fat.—The dry butter from the water determination is dissolved in the dish with *absolute* ether, or with 76° benzine. The contents of the dish are then transferred to a weighed Gooch filter with the aid of a wash bottle filled with the solvent, and are washed till free from fat. The crucible and contents are heated at the temperature of boiling water till the weight is constant. The weight of fat is calculated from the data obtained.

Substitute method for fat.—Water may be determined by drying the butter on asbestus or sand, and the fat extracted by *anhydrous alcohol-free* ether[1] in an extraction apparatus. The extract, after evaporation of the ether, is heated to constant weight at the temperature of boiling water and weighed.

Determination of casein and ash.—The crucible containing the residue from the fat determination, consisting of casein and ash, is covered and heated, gently at first, gradually raising the temperature to just below red heat. The cover may then be removed and the heat continued till the contents of the crucible are white. The loss in weight of the crucible and contents represents casein, and the residue in the crucible mineral matter. In this mineral matter, dissolved in water slightly acidulated, chlorine may be determined gravimetrically with silver nitrate, or volumetrically, using potassium chromate as an indicator.

ESTIMATION OF SALT (NaCl).

Volumetric method.—The amount of the butter or butter substitute to be taken is from 5 to 10 grs.; weigh in a counterpoised beaker-glass. The butter (fresh from the refrigerator) is placed, in portions of about 1 gr. at a time, in the beaker, these portions being taken from different parts of the sample. By this means a reasonably fair sample of the whole is obtained.

The given quantity having been weighed out, it is removed from the pan.

Hot water is now added (about 20 cc.) to the beaker containing the butter, and after it has melted, the liquid is poured into the bulb of a separating funnel. The stopper is now inserted and the contents shaken for a few moments.

After standing until the fat has all collected on top of the water, the stopcock is opened, and the water, containing most of the salt, is allowed to run into an Erlenmeyer flask, being careful to let none of the fat globules pass.

Hot water is again added to the beaker and thence poured into the separatory funnel, which is then well shaken, and the foregoing process is repeated from ten to fifteen times, using each time 10 to 20 cc. of water.

The resulting washings contain all but a mere trace of the NaCl orignally present in the butter.

Estimation of NaCl in filtrate.—The chloride of sodium is now determined in the filtrate by a standard solution of $AgNO_3$, using a few drops of a saturated solution of potassium chromate as an indicator.

SPECIFIC GRAVITY.

Weight of flask.—Use a small specific-gravity flask of from 25 to 30 cc. capacity. The stopper should be beveled to a fine edge on top and the lower end should be slightly concave to avoid any trapping of air. The flask is to be thoroughly washed with hot water, alcohol, and ether, and then dried for thirty minutes to one hour at 100° C. After cooling in a desiccator, the weight of the flask and stopper is accurately determined.

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[1] For the preparation of anhydrous alcohol-free ether, see under ether extract in methods for the analysis of cattle foods p. 189.

Weight of water.—The flask in an appropriate holder (Fig. 1), conveniently made of galvanized iron, is filled with freshly boiled and still hot distilled water, and placed in a bath of pure, very hot, distilled water in such a way that it is entirely surrounded by the liquid with the exception of the top.

The water of the bath is kept in brisk ebullution for thirty minutes, any evaporation from the flask being replaced by the addition of boiling distilled water. The stopper[1] is then inserted, the flask removed, wiped dry, and after it is nearly cooled

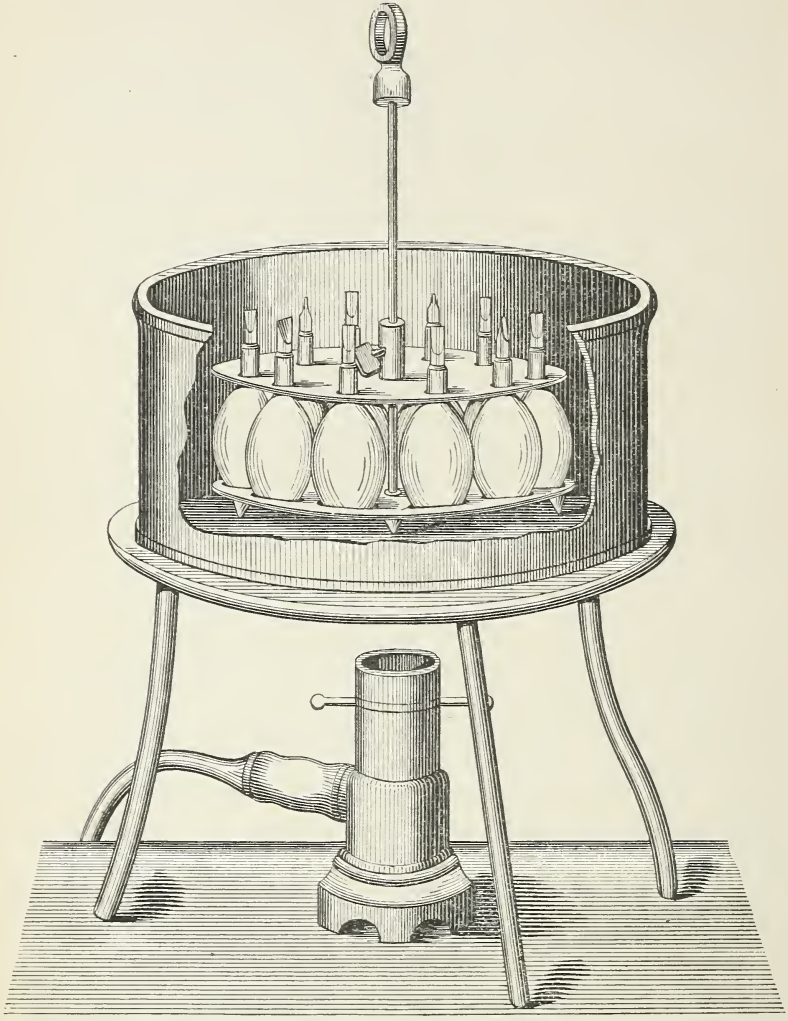

FIG. 1.

to room temperature, placed in the balance, and weighed when balance temperature is reached. A convenient size of holder will enable the analyst to use eight flasks at once. The temperature at which water boils in each locality may also be determined; but unless at very high altitudes, or on days of unusual barometric disturbance, the variations will not be great, and will not appreciably affect the results.

[1] The stopper should be kept for a few minutes before use in hot distilled water.

ALTERNATE METHOD OF ESTIMATING THE WEIGHT OF WATER IN FLASKS.

Computation from the volume of water in flask at a given temperature.

Formula for calculating the volume V, in cubic centimeters, of a glass vessel, from the weight P of water of temperature t contained therein, and the volume V′ at any other temperature t' (from Landolt and Börnstein's physical-chemical tables, p. 39, Table 17):

$$V = P\frac{p}{d}$$

$$V' = P\frac{p}{d}\left[1 + \gamma\,(t' - t)\right]$$

p = weight (in brass weights) of 1 cc. of H_2O in vacuo; see formula, Table 3. (This is so nearly 1, that it will not affect the result in the fifth place of decimals, and may therefore be disregarded.) Hence the formula stands:

$$V' = P\frac{1}{d}\left[1 + \gamma\,(t' - t)\right]$$

d = density of water at temperature t (see Table 12).

γ = .000025, the cubical expansion coefficient of glass.

From this volume the weight of the water may be readily obtained by referring to Tables 13 and 13*a*.

Weight of fat at the temperature of boiling water.—The flask is emptied of its water, rinsed with alcohol and ether, and dried again for a few minutes at 100° C. It is then filled with the dry, hot, fresh-filtered fat, which should be entirely free from air bubbles.

The stoppered flask is then replaced in the water bath, kept for thirty minutes at the temperature of boiling water, removed, and treated as above. The weight of fat having been determined, the specific gravity is obtained by dividing it by the weight of water previously found.

Example.

	Grams.
Weight of flask No. 22, dry	10.0197
Weight of flask No. 22, plus water	37.3412
Weight of water	27.3215
Weight of flask No. 22, plus fat	34.6111
Weight of fat	24.5914

Specific gravity = 24.5914 ÷ 27.3215 = .90008.

The weight of the flask dry and empty, and the weight of water at 99° C. to 100° C. contained therein, may be used constantly if great care be taken in handling and cleaning the apparatus.

Example.

	Grams.
Weight of flask No. 10, dry and empty	10.0028
Weight of flask after three weeks' use	10.0030

MELTING POINT.

The apparatus for determining the melting point (shown in Fig. 2) consists of (1) an accurate thermometer for reading easily tenths of a degree; (2) a cathetometer for reading the thermometer (this may be done with an eyeglass if held steadily and properly adjusted); (3) a thermometer; (4) a tall beaker glass 35 centimeters high and 10 centimeters in diameter; (5) a test tube 30 centimeters long and 3.5 centimeters in diameter; (6) a stand for supporting the apparatus; (7) some method of stirring the water in the beaker; for example, a blowing bulb of rubber, and a bent glass tube extending to near the bottom of the beaker; (8) a mixture of alcohol and water of the same specific gravity as the fat to be examined.

The disks of the fat are prepared as follows: The melted and filtered fat is allowed to fall from a dropping tube, from a height of 15 to 20 centimeters, onto a smooth piece of ice floating in water. The disks thus formed are from 1 to 1.5 centimeters in diameter, and weigh about 200 milligrams. By pressing the ice under the water, the disks are made to float on the surface, whence they are easily removed with a steel spatula, which should be cooled in the ice water before using.

The mixture of alcohol and water is prepared by boiling distilled water, and 95 per cent alcohol, for ten minutes to remove the gases which they may hold in solution. While still hot, the water is poured into the test tube already described until it is nearly half full. The test tube is then nearly filled with the hot alcohol. It should

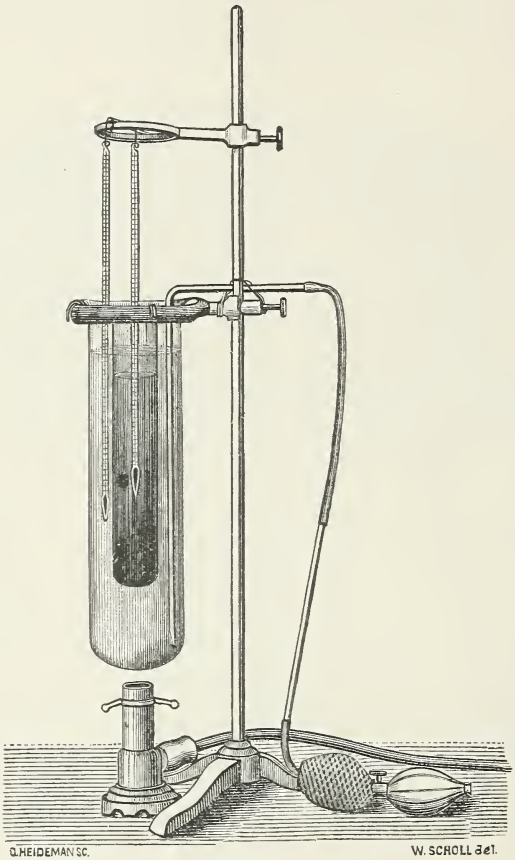

FIG. 2.

be poured in gently down the side of the inclined tube to avoid too much mixing. If the tube is not filled until the water has cooled, the mixture will contain so many air bubbles as to be unfit for use. These bubbles will gather on the disk of fat as the temperature rises, and finally force it to the top of the mixture.

The test tube containing the alcohol and water is placed in a tall beaker containing water and ice. The disk of fat is dropped into the tube from the spatula, and at once sinks until it reaches a part of the tube where the density of the alcohol-water is exactly equivalent to its own. Here it remains at rest, and free from the action of any force save that inherent in its own molecules.

The delicate thermometer is placed in the test tube, and lowered until the bulb is just above the disk. In order to secure an even temperature in all parts of the alcohol mixture in the vicinity of the disk, the thermometer is moved from time to time in a circular pendulous manner. A tube prepared in this way will be suitable for use for several days; in fact until the air bubbles begin to attach themselves to the disk of fat. The two liquids will not become sufficiently mixed to lose the property of holding the disks at a fixed point, even when they have been kept for several weeks.

Still, in practice, owing to the absorption of air, it has been found necessary to prepare new solutions every third or fourth day.

The disk having been placed in position, the water in the beaker glass is slowly heated, and kept constantly stirred by means of the blowing apparatus already described.

When the temperature of the alcohol-water mixture rises to about 6 degrees below the melting point, the disk of fat begins to shrivel, and gradually rolls up into an irregular mass.

The thermometer is now lowered until the fat particle is even with the center of the bulb. The bulb of the thermometer should be small, so as to indicate only the temperature of the mixture near the fat. A gentle rotatory movement should be given to the thermometer bulb, which might be done with some kind of clockwork. The rise of temperature should be so regulated that the last 2 degrees of increment require about ten minutes. The mass of fat gradually approaches the form of a sphere, and when it is sensibly so, the reading of the thermometer is to be made. As soon as the temperature is taken, the test tube is removed from the bath and placed again in the cooler. A second tube, containing alcohol and water, is at once placed in the bath. The test tube (ice water being used as a cooler) is of low enough temperature to cool the bath sufficiently. After the first determination, which should be only a trial, the temperature of the bath should be so regulated as to reach a maximum about 1.5 degrees above the melting point of the fat under examination.

Working thus with 2 tubes, about three determinations can be made in an hour. After the test tube has been cooled, the globule of fat is removed with a small cup attached to a wire, before another disk of fat is put in.

The distilled water for floating the piece of ice on which the disks are made, should be recently boiled, to free it of all air particles. It is important that the disks be free from air bubbles.

The edge of the disks should not be allowed to touch the sides of the tube. This accident rarely happens, but in case it should take place, and the disk adhere to the sides of the tube, a new trial should be made.

Triplicate determinations should be made, and the second and third results should show a near agreement.

Example.

Melting point of sample of butter 6372 (No. 3):

	Degrees C.
(1)	33.15
(2)	33.05
(3)	33.00

ESTIMATION OF VOLATILE ACIDS.

Reagents.

SOLUTION OF CAUSTIC SODA.

1. 100 grs. of NaOH dissolved in 100 cc. of pure water. The caustic soda should be as free as possible from carbonates, and be preserved from contact with the air.

2. Alcohol, of about 95 per cent, redistilled with caustic soda.

3. Solution of sulphuric acid containing 25 cc. of strongest H_2SO_4 in 1,000 cc. of water.

4. An accurately standardized approximately decinormal solution of barium hydrate.

5. Alcoholic solution of phenol-phthalein.

APPARATUS.

1. Saponification flasks of hard, well-annealed glass, capable of resisting the tension of alcohol vapor at 100° C. The flask shown in Fig. 3 is most suitable for this purpose. It should have from 250 to 300 cc. capacity.

Instead of such a flask, an Erlenmeyer flask of the same capacity fitted with a long glass tube or reflux condenser may be used.

2. A pipette graduated to deliver 40 cc.

3. Distilling apparatus as represented in Fig. 4. One with a smaller bulb tube may also be used.

4. An accurately calibrated burette reading to tenths of a cubic centimeter.

THE MANIPULATION.

Weighing the fat.—The butter or fat to be examined should be melted, and kept in a dry warm place at about 60° C. for two or three hours, until the moisture and curd have entirely settled out. The clean supernatant fat is poured off and filtered through a dry filter paper in a jacketed funnel containing boiling water, to remove all foreign matter and any traces of moisture. Should the filtered fat in a fused state not be perfectly clear, the treatment above mentioned must be repeated.

The saponification flasks are prepared by having them thoroughly washed with water, alcohol, and ether, wiped perfectly dry on the outside, and heated for one hour to 100° C. (*circa*). The flasks should then be placed in a tray by the side of the balance and covered with a silk handkerchief until they are perfectly cool. They must not be wiped with a silk handkerchief within fifteen or twenty minutes of the time they are weighed. The weight of each flask is determined accurately, using a flask for a counter-poise, or dispensing with the same, as may be convenient. The weight of the flasks having been accurately determined, they are charged with the melted fat in the following way:

A pipette with a long stem marked to deliver 5.75 cc. is warmed to a temperature of about 50° C. The fat having been poured back and forth once or twice into a dry beaker in order to thoroughly mix it, is taken up in the pipette, the nozzle of the pipette carried to near the bottom of the flask, having been previously wiped to remove any adhering fat. The 5.75 cc. of fat are allowed to flow into the flask, and the pipette is removed. After the flasks have been charged in this way, they should be re-covered with the silk handkerchief and allowed to stand fifteen or twenty minutes, when they are again weighed.[1]

The saponification.—10 cc. of 95 per cent alcohol redistilled from caustic soda are added to the fat in the flask, and then 2 cc. of the concentrated soda solution; a soft cork stopper is now inserted in the flask, and tied down with a piece of twine. The saponification is then completed by placing the flask upon the water or steam bath as indicated in Fig. 3. The flask during the saponification, which should last one hour, should be gently rotated from time to time, being careful not to project the soap for any distance up the sides of the flask. At the end of an hour the flask, after having been cooled to near the room temperature, is opened.

[1] Before weighing the flasks, any desiccating material used in the balance should be removed. If round-bottom flasks are employed, a special form of holder must be used. This is made on the principle of a test tube rack, the lower board of the tray being perforated so as to receive the round bottom of the flask, and being protected by lugs, so that on being placed on a table, the bottoms of the flasks do not touch the table.

Example:

	Grams.
Weight of counter-poised flask No. 3	22.5904
Weight of counter-poised flask No. 3 + fat	27.6734
Weight of fat	5.0830

Removal of the alcohol.—The stoppers having been laid loosely in the mouth of the flask, the alcohol is removed by dipping the flask into a steam bath. The steam should cover the whole of the flask except the neck. After the alcohol is nearly removed, frothing may be noticed in the soap, and to avoid any loss from this cause or any creeping of the soap up the sides of the flask, it should be removed from the bath and shaken to and fro until the frothing disappears. The last traces of alcohol vapor may be removed from the flask by waving it briskly, mouth down, to and fro. Complete removal of the alcohol with the precautions above noted should take about forty-five minutes.

Dissolving the soap.—After the removal of the alcohol the soap should be dissolved by adding 100 cc. of recently boiled distilled water, warming on the steam bath with occasional shaking, until solution of the soap is complete.

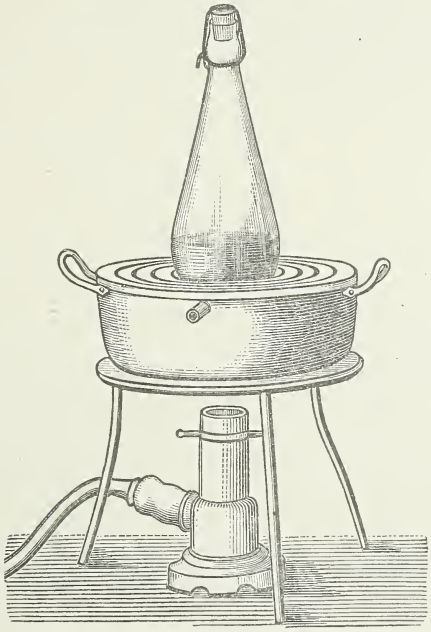

Fig 3.

Setting free the fatty acids.—When the soap solution has cooled to about 60° or 70° C., the fatty acids are separated by adding 40 cc. of the dilute sulphuric acid solution mentioned above.

Melting the fatty acid emulsion.—The flask should now be re-stoppered as in the first instance, and the fatty acid emulsion melted by replacing the flask on the steam bath. According to the nature of the fat examined, the time required for the fusion of the fatty acid emulsions may vary from a few minutes to several hours.

The distillation.—After the fatty acids are completely melted, which can be determined by their forming a transparent oily layer on the surface of the water, the flask is cooled to room temperature, and a few pieces of pumice stone added. The pumice stone is prepared by throwing it, at a white heat, into distilled water, and keeping it under water until used. The flask is now connected with a condenser (Fig. 4), slowly heated with a naked flame until ebullition begins, and then the distillation continued by regulating the flame in such a way as to collect 110 cc. of the distillate in, as nearly as possible, thirty minutes. The distillate should be received in a flask accurately graduated at 110 cc.

Titration of the volatile acids.—The 110 cc. of distillate, after thorough mixing, is filtered through perfectly dry filter paper, and collected in a flask graduated at 100 cc. The 100 cc. of the filtered distillate is poured into a beaker holding from 200 to 250 cc., 0.5 cc. phenol-phthalein solution added, and decinormal barium hydrate run in until a red color is produced. The contents of the beaker are then returned to the measuring flask to remove any acid remaining therein, poured again into the beaker, and the titration continued until the red color produced remains apparently unchanged for two or three minutes.

ALTERNATE METHOD OF DETERMINING VOLATILE ACIDS.

SAPONIFICATION WITHOUT THE USE OF ALCOHOL.

To avoid the danger of loss from the formation of ethers, and the trouble of removing the alcohol after saponification, the fat may be saponified with a solution of caustic potash in a closed flask without alcohol. The operation is carried on exactly as indicated above for saponification in a closed flask, using caustic potash solution

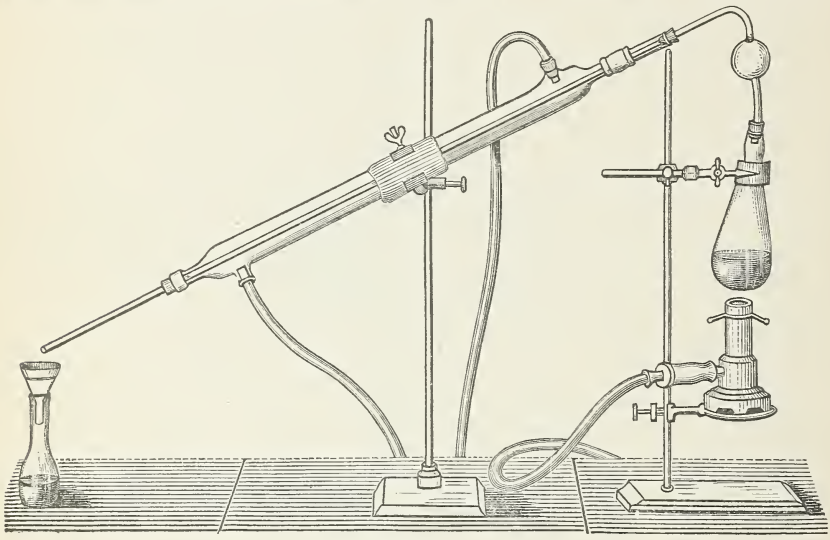

FIG. 4.

instead of soda, and omitting the operation for volatilizing the alcohol. The caustic potash is prepared as follows: Dissolve 100 grs. of the purest potassium hydrate in 58 grs. of hot distilled water. Allow to cool in a stoppered vessel, decant the clear caustic solution, and preserve in a vessel out of contact with the air. For the saponification use 2 cc. of the caustic potash solution, which is poured on the fat after it has solidified in the flask. Great care must be taken that none of the fat is allowed to rise on the sides of the saponifying flask to a point where it can not be reached by the alkali. During the process of saponification, the flask can only be very gently rotated, in order to avoid the difficulty mentioned. This process is not recommended in any except a closed flask with round bottom. In the subsequent solution of the soap use only 80 cc. of distilled water, and in setting free the fatty acids, use 60 cc. of the dilute sulphuric acid. In other respects the distillation is conducted as described. Potash is used instead of soda, so as to form a softer soap and thus allow a more perfect saponification.

The saponification may also be conducted as follows: The alkali and fat in the melted state are shaken vigorously in the saponification flask until a complete emulsion is secured. The rest of the operation is then conducted as above.

Iodine Equivalent.

REAGENTS.

1. Dissolve 25 grs. of pure iodine in 500 cc. of 95 per cent alcohol. Dissolve 30 grs. of mercuric chloride in 500 cc. of 95 per cent alcohol. The last solution, if necessary, is filtered, and then the two solutions mixed. The mixed solution should be allowed to stand twelve hours before using.

2. *Decinormal hyposulphite of sodium solution.*—Take 24.6 grs. of chemically pure hyposulphite of soda freshly pulverized as finely as possible and dried between filter or blotting paper. Make this up to 1,000 cc. at the temperature at which the titrations are to be made.

3. *Starch paste.*—1 gr. of starch boiled in 200 cc. of distilled water for ten minutes and cooled to room temperature.

4. *Solution of iodide of potassium.*—150 grs. of iodide of potassium dissolved in water and made up to 1 liter.

5. *Solution of bichromate of potassium.*—Dissolve 3.874 grs. of chemically pure bichromate of potassium in distilled water, and make the volume up to 1 liter at the temperature at which the titrations are to be made.

MANIPULATION.

Standardizing the hyposulphite of sodium solution.—Run 20 cc. of the bichromate of potassium solution to which has been added 10 cc. of the solution of iodide of potassium, into a glass-stoppered flask. Add to this 5 cc. of strong hydrochloric acid. Allow the solution of hyprosulphite of sodium to flow slowly into the flask until the yellow color of the liquid has almost disappeared. Add a few drops of the starch paste and with constant shaking continue to add the hyposulphite of sodium solution until the blue color just disappears. The number of cubic centimeters of hyposulphite solution used multiplied by 5 is equivalent to 1 gr. of iodine.

Example.—20 cc. $K_2Cr_2O_7$ solution required 16.2 cc. hyposulphite of soda; then $16.2 \times 5 = 81 =$ number cubic centimeters of hyposulphite solution equivalent to 1 gr. of iodine. Then 1 cc. hyposulphite solution = 0.0124 gr. of iodine. Theory for decinormal solution of hyposulphite of sodium, 1 cc. = 0.0127 gr. of iodine.

Weighing the sample.—About 1 gr. of butter fat is to be weighed in a glass-stoppered flask holding about 300 cc., with the precautions mentioned for weighing the fat for determining volatile acids.

Absorption of iodine.—The fat in the flask is dissolved in 10 cc. of chloroform. After complete solution has taken place, 30 cc. of the iodine-mercuric chloride solution is added. The flask is now placed in a dark place and allowed to stand, with occasional shaking, for three hours.

Titration of the unabsorbed iodine.—100 cc. of distilled water is added to the contents of the flask, together with 20 cc. of the iodide of potassium solution. Any iodine which may be noticed upon the stopper of the flask should be washed back into the flask with the iodide of potassium solution. The excess of iodine is now taken up with the hyposulphite of sodium solution, which is run in gradually, with constant shaking, until the yellow color of the solution has almost disappeared. A few drops of starch paste are then added, and the titration continued until the blue color has entirely disappeared. Toward the end of the reaction the flask should be stoppered and violently shaken, so that any iodine remaining in solution in the chloroform may be taken up by the iodide of potassium solution in the water. A sufficient quantity of hyposulphite of sodium solution should be added to prevent a reappearance of any blue color in the flask for five minutes.

Setting the value of the iodine solution by the hyposulphite solution.—At the time of adding the iodine solution to the fats, two blank flasks of the same size and nature as those used for the determination should be employed for conducting the operation

described above, without the presence of any fat. In every other respect the performance of the blank experiments should be just as described. These blank experiments must be made each time the iodine solution is used.

EXAMPLE.

BLANK DETERMINATIONS.

(1) 30 cc. iodine solution required 46.4 cc. hyposulphite of sodium solution.
(2) 30 cc. iodine solution required 46.8 cc. of hyposulphite of sodium solution.
Mean 46.6.

PERCENTAGE OF IODINE ABSORBED.

Weight of fat taken	grams.	1.0479
Quanty of iodine solution used	cubic centimeters.	0.39
Hyposulphite equivalent to iodine used	do	46.6
Hyposulphite equivalent to remaining iodine	do	14.7
Hyposulphite equivalent to iodine absorbed	do	31.9

Percentage of iodine absorbed, $31.9 \times 0.0124 \times 100 \div 1.0479 = 37.75$ per cent.

ELECTIVE DETERMINATIONS.

For the further study of the properties of butter the following recommendations are made, to be followed by the analyst, or not, as he may elect:

Reduction of silver.—To determine whether or not the butter has been derived from a cow fed on cotton-seed meal, it will usually be sufficient to test the sample by Bechi's reagent.

The solutions used are as follows: 5 grs. of silver nitrate is dissolved in 100 cc. of 95 per cent alcohol, and 1 cc. of strong nitric acid added. The manipulation is conducted as follows: Measure out 5 cc. of the fat into a small porcelain dish and add 5 cc. of the above reagent. Stir together thoroughly with a stirring rod and place upon the steam bath for ten minutes. Notice the changes of color which take place, the intensity of the color produced, and the amount of silver deposited as a mirror on the sides of the dish.

Reduction test applied to the fatty acids.—Instead of testing the original glycerides as above, the test may be applied to the fatty acids in accordance with the following directions:

Reagent.—Dissolve 30 grs. of nitrate of silver in 100 cc. of water.

Manipulation.—Place 5 cc. of the fatty acid in the porcelain dish which is used above, and dissolve in 20 cc. of 95 per cent alcohol. Add 2 cc. of the reagent, stir thoroughly together, and heat on the water bath as in the first instance.

Melting point of the fatty acids.—The melting point of the fatty acids may be determined as described in Bulletin No. 13, Chem. Div. U. S. Dept. of Agr., Part 4, p. 448.

The melting point of the fatty acids can not be determined in the same apparatus and by the same methods as those described for the fats themselves, because the acids are soluble in alcohol. It should be remembered that the melting point of the fatty acids is slightly above that of the glycerides, and the first determination in every case should be solely for the purpose of determining approximately the temperature at which the fat melts.

The bulb of a delicate thermometer is coated with the fatty acid, and the thermometer by means of a cork is fastened in a round flask of 250 cc. capacity. The bulb of the instrument should occupy as nearly as possible the center of the flask. The cork should have an air passage for the equalization of the pressure. The flask is slowly heated in a current of warm air, or otherwise, and as the melting point is approached a rotatory motion is given to it. When the fat melts it is seen to collect in a small drop on the lowest part of the bulb, remaining stationary while the flask is turned. The thermometer is best held horizontally.

Crystallizing point of the fatty acids.—The crystallizing point of the fatty acid is determined by means of the method described in Bulletin No. 13, Chem. Div. U. S. Dept. Agr., Part 4, p. 447.

A very delicate thermometer with a long bulb is used, the thermometer being graduated to tenths of a degree; the readings of the mercury are made with a small eyeglass. A test tube about 15 cm. in length and 2.5 to 3 cm. in diameter is filled with the melted fatty acids. The temperature at which the acids are melted should be sufficiently high to secure a complete liquefaction. The tube containing the fat is placed in a stopper carried in a bottle, so that the whole of the fatty acids may be contained in that part of the tube protected from external currents of air by the bottle. The bottom of this protected bottle should be warm, so that its temperature may be several degrees higher than the crystallizing point of the fatty acids. This precaution is necessary to avoid a too rapid crystallization of the fatty acids in the bottom of the test tube, and to secure as nearly as possible a uniform crystallization throughout the whole mass. The thermometer is suspended in such a manner that the bulb may occupy as nearly as possible the center of the fatty mass. The thermometer should be protected from currents of air, and should be kept perfectly still. The position of the mercury in the thermometer is carefully watched by the attendant as it gradually sinks toward the crystallizing point. When the crystals of the acids begin to appear in the bottom and on the sides of the test tube, the descent of the mercury will become very slow and finally cease. The lowest point reached by the mercury should be noted. As the crystallization extends inward toward the bulb of the thermometer, a point will be reached when the mercury will begin to rise. At that point, the test tube being held by the left hand, the thermometer should be taken by the right hand of the operator, and the partially crystallized mass of fat thoroughly stirred by turning the thermometer three or four times around the tube in both directions. Care should be exercised that at the end of this operation the bulb of the thermometer should hang as near as possible in the center of the crystallizing mass. Directly the above operation is accomplished the mercury will be seen to rise, and this rise of temperature will continue for some time, after which the mercury will remain stationary for one or two minutes. The highest point reached is taken as the true temperature of crystallization.

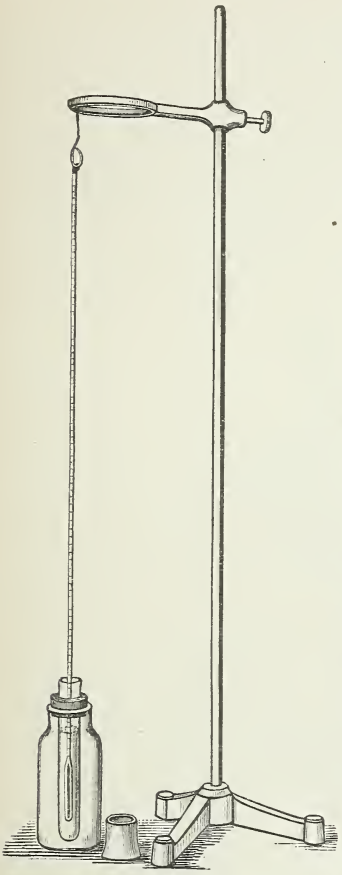

FIG. 5.

Refractive index.—The refractive index is conveniently determined by the apparatus of Prof. Abbe, Fig. 6. The manipulation is conducted as follows: Fine tissue paper of rather heavy body is cut into rectangular pieces 3 centimeters in length by 1.5 centimeters in breadth. One of these pieces of paper is placed on the lower of the two glass prisms of the apparatus. Two or three drops of the oil or fat are placed upon the paper, and the upper prism carefully placed in position so as not to move the paper from its place. In charging the apparatus with the oil in this way it is placed in the horizontal position. After the paper disk holding the fat is secured by replacing the upper prism, the apparatus is placed in its normal position, and the index

moved until the light directed through the apparatus by the mirror shows the field of vision divided into dark and light portions. The dispersion apparatus is now turned until the rainbow colors on the part between the dark and light field have disappeared. Before doing this, however, the telescope, the eyepiece of the apparatus, is so adjusted as to bring the cross lines of the field of vision distinctly in focus. The index of the apparatus is now moved back and forth until the dark edge of the field of vision falls exactly in the intersection of the cross lines. The refractive index of the fat under examination is then read directly upon the scale by means of a small magnifying glass. To check the accuracy of the first reading, the dispersion apparatus should be turned through an angle of 180° until the colors have again disappeared, and the scale of the instrument again read. These two readings should nearly coincide, and their mean is the true reading of the fat under examination.

For butter fats the apparatus should be kept in a warm place, the temperature of which does not fall below 30° C. For reducing the results obtained to a standard temperature, say 25° C., the following factor may be used. The mean rate of variation

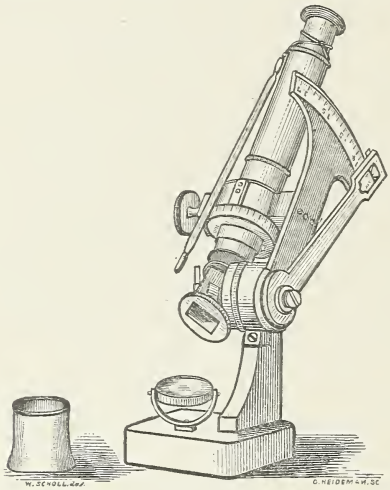

Fig. 6.

in the refractive index of butter fats for each degree of temperature was determined by me in 12 samples of butter fat (see Jour. Anal. and Applied Chem., Vol. 2, Part 3, July, 1888). This factor is .000176. As the temperature rises the refractive index falls.

Example.

Refractive index of a butter fat determined at 32.° 4 = 1.4540, reduced to 25° C. as follows: 32.4 — 25 = 7.4 = 0.0013; then 1.4540 + 0.0013 = 1.4553.

The instrument used should be set with distilled water at 25° C., the theoretical refractive index of water at that temperature being 1.3330. In the determination above given, the refractive index of pure water measured 1.3300; hence the above numbers should be corrected for theory by the addition of 0.0030, making the corrected index of the butter fat mentioned at the temperature given 1.4583.

Soluble and insoluble acids.—Use method described in Bulletin 13, Chem. Div. U. S. Dept. of Agr., Part 4, pp. 455 to 461.

Saponification equivalent.—Use method described in Bulletin 13, Chem. Div. U. S. Dept. of Agr., Part 4, pp. 461, 462.

Rise of temperature with sulphuric acid.—Use method described in Bulletin 13, Chem. Div. U. S. Dept. of Agr., Part 4, pp. 443–446.

METHODS FOR MILK ANALYSIS.

WATER.

Evaporate 1 to 2 grs. of milk in a tared flat dish containing from 15 to 20 grs. of pure dry sand, or without sand, until apparently dry. Transfer to an air bath, and dry for one hour at the temperature of boiling water. Cool in a desiccator, and weigh rapidly to avoid absorption of hygroscopic moisture.

TOTAL NITROGENOUS MATTER.

Place in a Kjeldahl digestion flask a known weight (about 5 grs.) of milk, and proceed, without evaporation, exactly as described for this method by the nitrogen reporter.

FAT; METHOD OF ADAMS, MODIFIED BY WILEY.

Coils made of thick filter paper, cut into strips 2.5 by 25 inches, are thoroughly extracted with ether and alcohol, or the weight of the extract corrected by a constant obtained for the paper; if this latter method is used, a small amount of anhydrous sodium carbonate should be added. About 5 cc. of the well-mixed milk is placed in a small beaker, covered with a watch glass, and weighed. From a weighing bottle about 5 grs. of milk is transferred to the coil by a pipette, care being taken to keep the end of the coil held in the fingers dry. The coil, dry end down, on a piece of glass, is dried at the temperature of boiling water for one hour, or, better, dried in hydrogen at the temperature of boiling water, transferred to a siphon extraction apparatus, and extracted by at least 12 siphonings with absolute ether or petroleum spirit boiling at about 45° C. The extracted fat is dried in hydrogen and weighed.

If the milk is sour, add about 10 per cent, by weight, of strong ammonia water, and a small quantity of anhydrous Na_2CO_3, correcting the results for the ammonia added.

ALTERNATE METHOD OF ESTIMATING WATER AND FAT IN MILK.

Method of Babcock.—In the bottom of a perforated test tube is placed a plug of clean cotton, the tube is then filled three-quarters full of ignited asbestos, lightly packed, and a plug of cotton inserted over it. The tube and contents are weighed, the plug of cotton carefully removed, 5 grs. of milk from a weighed pipette run into it, and the plug of cotton replaced. The tube, connected at its lower end by a rubber tube and adapter with a filter pump, is placed in a drying oven at a temperature of 100° C., and a slow current of dry air drawn through it until the water is completely expelled, which in no case requires more than two hours.

The tube containing the solids from the above operation is placed in an extraction apparatus and exhausted with ether in the usual way.

THE ESTIMATION OF SUGAR.

The reagents, apparatus, and manipulation necessary to give the most reliable results in milk-sugar estimation are as follows:

Reagents.—(1) *Basic plumbic acetate*, specific gravity 1.97. Boil a saturated solution of sugar of lead with an excess of litharge, and make it of the strength indicated above. 1 cc. of this will precipitate the albumins in 50 to 60 cc. of milk.

(2) *Acid mercuric nitrate.* Dissolve mercury in double its weight of nitric acid, specific gravity 1.42. Add to the solution an equal volume of water. 1 cc. of this reagent is sufficient for the quantity of milk mentioned above. Larger quantities can be used without affecting the results of polarization.

(3) *Mercuric iodide with acetic acid.* KI 33.2 grs., $HgCl_2$ 13.5 grs., $C_2H_4O_2$ 20 cc., H_2O 64 cc.

Apparatus.—(1) Pipettes marked at 59.5, 60, and 60.5 cc. (2) Sugar flasks marked at 102.4 cc. (3) Filters, observation tubes, and polariscope. (4) Specific gravity spindle and cylinder. (5) Thermometers.

Manipulation.—(1) The room and milk should be kept at a constant temperature. It is not important that the temperature should be of any given degree. The work can be carried on equally well at 15° C., 20° C., or 25° C. The slight variations in rotatory power within the above limits will not affect the result for analytical purposes. The temperature selected should be the one which is most easily kept constant.

(2) The specific gravity of the milk is determined. For general work this is done by a delicate specific gravity spindle. Where greater accuracy is required use specific gravity flasks.

(3) If the specific gravity be 1.026, or nearly so, measure out 60.5 cc. into the sugar flask. Add 1 cc. of mercuric nitrate solution, or 30 cc. of mercuric iodide solution, and fill to the 102.4 cc. mark. The precipitated albumin occupies a volume of about 2.44 cc. Hence the milk solution is really 100 cc If the specific gravity is 1.030, use 60 cc. of milk. If the specific gravity is 1.034, use 59.5 cc. of milk.

(4) Fill up to the mark in the 102.4 cc. flask, shake well, filter, and polarize.

NOTES.—In the above method of analysis the specific rotatory power of milk sugar is taken at 52.5, and the weight of it in 100 cc. solution to read 100 degrees in the cane-sugar scale at 20.56 grs. This is for instruments requiring 16.19 grs. sucrose to produce a rotation of 100 sugar degrees. It will be easy to calculate the number for milk-sugar, whatever instrument is employed.

Since the quantity of milk taken is three times 20.56 grs., the polariscopic readings divided by 3 give at once the percentage of milk sugar when a 200-millimeter tube is used.

If a 400-millimeter tube is employed, divide the reading by 6; if a 500-millimeter tube is used, divide by 7.5.

Since it requires but little more time, it is advisable to make the analysis in duplicate, and take four readings for each tube. By following this method gross errors of observation are detected and avoided.

By using a flask graduated at 102.4 for 60 cc. no correction for volume of precipitated casein need be made. In no case is it necessary to heat the sample before polarizing.

ALTERNATE METHOD.

The sugar may also be determined, either gravimetrically or volumetrically, by alkaline copper solution.

ESTIMATION OF ASH.

In a weighed dish put 20 cc. of milk from a weighing bottle; add 6 cc. of HNO_3, evaporate to dryness, and burn at low red heat until ash is free from carbon.

METHODS OF ANALYSIS OF FERMENTED LIQUORS.

I. *Specific gravity.*—This determination is made with a picnometer, or a Westphal balance controlled by a picnometer, at a temperature of 15.5° C.

II[1]. *Alcohol.*—100 cc. of the liquor measured at 15.5° C, to which 50 cc. of water has been added, is distilled until 100 cc. at 15.5° C. has come over. The specific gravity of the distillate is taken at 15.5° C., and the percentage of alcohol determined by reference to alcohol tables.

III. *Extract.*—50 cc. (weighed), or in case of sweet wines a smaller amount, is evaporated on a water bath in a platinum dish 85 millimeters in diameter and of about 75 cc. content. The residue is heated for two and one-half hours in a drying oven at 100°.

[1] The method for alcohol adopted by the association was as follows: "100 cc. of wine at 25° distilled, preferably in glass."

This imperfect description of a method is the cause of the insertion of the above inofficial substitute.—H. W. W.

In sweet wines, *i. e.*, those containing more than 0.5 gr. sugar in 100 cc., less than 50 cc. should be taken and diluted to 50 cc. with water.

IV. *Acidity* (total acid constituents of the wine expressed as tartaric acid).—If carbonic acid is present, expel by shaking. Titrate with dilute alkali solution. The neutral point is determined by adding a drop of the solution to delicate litmus paper.

V. *Volatile acids* (expressed as acetic acid).—Distill in a current of steam, and titrate the carefully condensed distillate with standard alkali (decinormal).

VI. *Glycerine.*—(1) This is detemined in dry wines as follows: The alcohol is driven off from 100 cc. of wine, lime or magnesia is added, and then evaporated to dryness. The residue is boiled with 90 per cent. alcohol, filtered, and the filtrate evaporated to dryness. This residue is dissolved in 10 to 20 cc. of alcohol, 15 to 30 cc. of ether is added, and the mixture allowed to stand until it is clear. It is then decanted from the sticky precipitate into a glass-stoppered weighing bottle, evaporated to constant loss of weight, and weighed.

(2) The following method is employed for sweet wines: 100 cc. of wine is measured into a porcelain dish and evaporated on the water bath to a sirupy consistency, mixed with 100 to 150 cc. of absolute alcohol, poured into a flask, ether added in the proportion of one and one-half volumes to each volume of alcohol used, the flask well shaken, and allowed to stand until the liquid becomes clear. This is then poured off, and the residue again treated with a mixture of alcohol and ether. The liquids are mixed, the alcohol and ether driven off, the residue dissolved in water, and treated as in (1).

(3) It is necessary to test the glycerine from sweet wines for sugar, and if any is present it must be estimated by Soxhlet's method, and its weight subtracted from that of the glycerine.

VII. *Sugar.*—This is to be determined by Sohxlet's method. The presence of unfermented cane sugar is to be shown by inversion.

The wine should not contain more than 1 per cent of sugar.

Preliminary test.—100 cc. of wine accurately neutralized with normal soda solution is dealcoholized by evaporation to two-thirds, and the original volume restored with water (red wines are decolorized with pure animal charcoal); 25 cc. of copper solution and 25 cc. of seignette salt solution are heated to boiling in a deep porcelain evaporating dish; wine is added from a burette till the fluid, after two minutes' boiling, no longer has a blue color. This gives an approximate estimate of the sugar present, and from this we can dilute the wine to very nearly 1 per cent of sugar.

Final determination.—25 cc. of wine, as in preliminary test, 25 cc. of seignette solution, and 23 to 24 cc. of diluted wine (approximately 1 per cent), are heated two minutes to boiling, the precipitated copper allowed to settle, and filtered through a double filter. If the filtrate is greenish, it contains copper; if the filtrate is yellow, it is acidulated, and after cooling a little acetic acid is added and two or three drops ferrocyanide of potassium. If a reddish color is formed, copper is still present, and a sufficient amount of sugar has not been added to reduce the 50 cc. of Fehling's solution. The experiment is repeated, adding 1 to 2 cc. more wine if copper was present in the filtrate; if no copper was in the filtrate, 1 cc. less of wine. The experiment is continued till filtrates are obtained containing a difference of 0.1 cc. of wine, one of which contains copper solution and one not. The mean of the two contains the sugar equivalent to 50 cc. of Fehling's solution; 50 cc. of Fehling's solution corresponds to 0.2375. gr. of grape sugar.

Polarization.—(1) The wine is decolorized with plumbic subacetate.

(2) A slight excess of sodic carbonate is added to the filtrate from (1). 2 cc. of a solution of plumbic subacetate are added to 40 cc. of white wine, and 5 cc. to 40 cc. of red wine, the solution is filtered, and 1 cc. of a saturated solution of sodic carbonate added to 21.0 or 22.5 cc. of the filtrate.

(3) The kind of apparatus used and the length of the tube are to be given, and the results estimated in equivalents of Wild's polari-strobometer with 200-millimeter tubes

(4) All samples rotating more than 0.5° to the right (in 220 millimeter tubes, after treating as above), and showing no change or but little change in their rotatory power after inversion, are to be considered as containing unfermented glucose (starch sugar) residue.

(5) Rotatory power of less than 0.3° to the right shows that impure glucose has not been added.

(6) Wines rotating between 0.3° and 0.5° to the right must be treated by the alcohol method.

(7) Wines rotating strongly to the left must be fermented, and their optical properties then examined.

VIII. *Tannin.*—Determine by the Neubauer and Löwenthal method.

100 cc. of wine is dealcoholized by continued boiling, and the lost weight restored by water. (1) 10 cc. of dealcoholized wine is transferred to a large porcelain evaporating dish, 1 liter of water added, also 10 cc. of dilute H_2SO_4, and then 20 cc. of indigo solution; titrate with $K_2Mn_2O_8$ solution, adding this reagent with great care (drop by drop) towards the end of the reaction. (2) 10 cc. of dealcoholized wine treated with bone black to remove tannin and coloring matter, after standing for a time, is filtered, washed with water and diluted to 1 liter, and titrated with $K_2Mn_2O_8$ solution, as in (1) (viz, after adding 10 cc. of dilute H_2SO_4 and 20 cc. of indigo).

From the number of cubic centimeters of $K_2Mn_2O_8$ used in (2) substract the number of cubic centimeters of $K_2Mn_2O_8$ required to oxidize the indigo, and the remainder will give the number of cubic centimeters of $K_2Mn_2O_8$ necessary to oxidize the matters other than tannin and color.

From the number of cubic centimeters of $K_2Mn_2O_8$ used in (1) take the value of the indigo used, and the remainder will be the number of cubic centimeters necessary to oxidize tannin, color, and other oxidizable matter. Subtract from this the number of cubic centimeters of $K_2Mn_2O_8$ from (2) after taking out the value of indigo, and the remainder will be the amount necessary to oxidize the tannin and color. (The amount required for color can be neglected.)

IX. *Potassic bitartrate.*—The determination of potassic bitartrate as such is to be omitted.

X. *Tartaric, malic, and succinic acids.*—(1) According to Schmidt and Hiepe's method.

To 200 cc. of wine evaporated one-half and allowed to cool, basic lead acetate is added to alkaline reaction. The precipitate is filtered off and washed with cold water till the filtrate shows a only slight lead reaction. The precipitate is washed from the filter with a wash bottle into a beaker and treated hot with H_2S till the lead is completely converted into sulphide, filtered hot, and washed with hot water. The filtrate and wash water are evaporated to 50 cc., neutralized with KHO, and again concentrated. An excess of Ca $(C_2H_2O_3)_2$ is added and allowed to stand 4 to 6 hours, with frequent stirring. It is then filtered and washed with water till the filtrate and wash water amount to 100 cc. The tartrate of lime is then converted into caustic lime by strong ignition, and, according to quantity, 10 to 15 cc. of normal HCl added, and the solution diluted and retitrated very accurately. For every cubic centimeter of normal HCl saturated by the caustic lime, 0.075 gr. of tartaric acid is to be set down, and to this 0.0286 gr. added, representing the total tartaric acid present in 200 cc. wine.

The filtrate from the tartrate of lime is now evaporated to about 20 to 30 cc., allowed to cool, and three times the volume of 96 per cent alcohol added. After standing several hours the precipitate is collected upon a weighed filter, dried at 100° C., and weighed. This represents the lime salts of the malic and succinic acids, and the tartaric acid still remaining in solution, and of the sulphuric acid in the wine. This precipitate is now dissolved in a minimum of HCl, washed with hot water, and then precipitated hot with K_2CO_3, and the $CaCO_3$ filtered off. The filtrate now contains the potash salts of the above-mentioned acids. This is neutralized with acetic acid and evaporated to a small volume, and precipitated hot with $BaCl_2$. This precipitate of succinate and sulphate of barium is treated on the filter with dilute HCl; the filtrate now contains only the succinate of barium, is treated with H_2SO_4 and the Ba precipi-

tated. (The $BaSO_4$ remaining on the filter is ignited and weighed.) The $BaSO_4$ obtained from the succinate solution is filtered off, washed, dried, ignited, and weighed; 223 parts of $BaSO_4$ correspond to 118 parts succinic acid. The succinic and sulphuric acids, as well as the tartaric acid remaining in solution, which was equal to 0.0286 gr., are to be calculated as lime salts, and subtracted from the total weight of the lime precipitate. The remainder is malate of lime, of which 172 parts correspond to 134 parts of malic acid.

(2) Determination of tartaric acid according to the modified Berthelot-Fleury method.[1]

50 cc. is measured, and 10 cc. of same neutralized with caustic potash. The two liquids are now mixed, and one-fifth of the mixture, 10 cc., is taken, and 50 cc. of a mixture of alcohol and ether added and allowed to stand twenty-four hours, and the separated cream of tartar filtered off, dissolved in water, and titrated. The excess of cream of tartar above that obtained in the estimation of tartar alone corresponds to the free tartaric acid.

XI. *Coloring matter.*—(1) Only anilin dyes are to be looked for.

(2) Special attention is to be paid to the spectroscopic behavior of rosanilin dyes as obtained by shaking wines with amylic alcohol before and after saturation with ammonia.

XII. *Inorganic matter* (ash).—Burn in the ordinary manner in a flat platinum dish at as low a heat as possible; repeated moistening, drying, and heating to redness are advisable to get rid of all organic substances.

XIII. *Citric acid.*—Presence to be shown by a qualitative test, as baric citrate.

XIV. *Sulphuric acid.*—To be determined in the wine after adding hydrochloric acid.

XV. *Chlorine.*—To be determined in the nitric acid solution of the burnt residue by Volhard's method.

XVI. *Lime, magnesia, and phosphoric acid.*—These are determined in the ash fused with sodic hydrate and potassic nitrate, the phosphoric acid by the molybdenum method.

XVII. *Pot ash.*—Either in the wine ash, as the platinum double salt, or in the wine itself, by Kayser's method.

XVIII. *Gums.*—Presence shown by precipitation with alcohol; 4 cc. of wine and 10 cc. of 96 per cent alcohol are mixed. If gum arabic has been added, a lumpy, thick, stringy precipitate is produced; whereas pure wine becomes at first opalescent and then floculent.

XIX. *Sulphurous acid.*—100 cc. of wine is distilled in a current of carbonic acid after the addition of phosphoric acid. The distillate carefully condensed is oxidized with bromine, and the amount of H_2SO_4 determined.

ABNORMAL CONSTITUENTS OF WINES.

Mannite.—When present in any considerable quantity, mannite can be detected by simply allowing the wine (a few drops will be sufficient) to evaporate spontaneously upon a glass slide or plate. The mannite will crystallize in a characteristic stellated form, which can be recognized at a glance. It should be allowed to stand twenty-four to forty-eight hours. When the quantity is small, extract the residue evaporated nearly or quite to dryness, with boiling 80 per cent alcohol. Evaporate this extract to dryness and extract with 95 per cent boiling alcohol; the mannite if present in any quantity will separate out in part upon cooling. If allowed to evaporate spontaneously, the characteristic stellated crystals can be obtained.

Lactic acid.—This will be found in the alcohol ether extract of the evaporated residue, and can be recognized by the usual tests. It may be determined by titration with standard alkali. It may be necessary to take larger quantities of the wine for this investigation.

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[1] This is the method furnished by the reporter; as given it is unintelligible.—H. W. W.

METHOD USED FOR THE APPROXIMATE DETERMINATION OF THE ALCOHOL AND SUGAR CONTENTS OF SWEET WINES BY U. S. INTERNAL-REVENUE OFFICERS.

The methods herewith described may briefly be stated to involve the determination of the specific gravity of the original liquid, of the extract, and of the distillate at 60° F., and from these three facts to ascertain, by reference to suitable tables, the alcohol and sugar contents (total solids) of the wine under examination.

Table I gives the correction to be applied to the readings of the saccharometer when taken at other than the normal temperature; Table II gives the specific gravity corresponding to the readings of the saccharometer at the normal temperature; Table III gives the specific gravity corresponding to the readings of the alcoholometer at the normal temperature, and Table IV gives the corrections to be applied to the readings of the alcoholometer when taken at other than the normal temperature.

Where the determinations are made by the officer at the vineyard, samples are taken direct from the vats or other vessels containing the wine, but when the determinations have to be made at the laboratory, then the officers are instructed to observe the following:

DIRECTIONS FOR SENDING SAMPLES FOR CHEMICAL ANALYSIS.

(1) All samples taken for analysis shall be not less than one quart each, put up in clean, strong, glass bottles. The officer taking the samples should *himself* thoroughly wash the bottles with hot water, rinsing them with cold water, and allowing them to drain dry, before taking the samples. The officer will see that the wine in the tank is thoroughly mixed and blended before the samples are taken.

(2) Rinse the bottles with the wine to be sampled, and then fill them to within an inch of the cork.

(3) A new and perfect cork should be used to close the neck of the bottle.

(4) Label each bottle with a gum label and state thereon: Date, name of producer, and location of winery; variety; serial number of package whence sample is taken by ———. The label will also be signed by the officer and owner, and will contain a statement to the effect that each signer agrees that the wine in the bottle to which the label is affixed is a fair average sample of the wine in the package described therein.

(5) Seal the cork with a distinctive seal and pack carefully in a box.

(6) Special care should be taken in sealing the samples and box, so that the identity may be readily established.

APPARATUS USED.

The outfit furnished internal-revenue officers consists of a set of 3 balling saccharometers reading in tenths, from 0–10, 10–20, 20–30 per cent; a set of 3 alcoholometers reading in tenths, from 0–10, 10–20, 20–30 per cent by volume (both kinds of hydrometers are tested by the Office of Weights and Measures, U. S. Coast and Geodetic Survey, and furnished with a table of corrections before they are given to officers); a chemical glass scale thermometer, Fahrenheit; glass measuring cylinders; a tin-lined copper still with block-tin condenser and alcohol lamp; a copper evaporating dish, shaped like a porcelain casserole, with tripod and alcohol lamp, and the necessary carrying cases.

Precautions to be observed in using the apparatus.—1. The apparatus (still, receiver, measuring cylinders, alcoholometer, and thermometer) must be clean and dry before they are used for a determination. For this purpose they will be thoroughly washed with clear water and wiped dry with a towel after each determination.

2. The temperature of the liquid under examination should always be brought as near as possible to the temperature at which the instruments are standardized, in order to reduce the sources of errors in the determination. For this purpose, where

the temperature of the liquid is higher than the standard (60° F.), the sample may be placed in a copper or thin glass cylinder, or other vessel, and surrounded with cold water until the temperature is sufficiently lowered; and, in case the temperature of the liquid is lower than the standard, a little tepid water may be used in which to place the cylinder. The corrections found in tables I and IV may, however, be applied when the temperature of the liquid is not above 70° F. or below 50° F.; in all other cases the temperature of the liquid under examination should be brought within these limits by the methods above given, before proceeding with the determination of the specific gravity by the hydrometer.

3. The distillation should be conducted at as low a temperature as possible, so as to avoid frothing or priming in the still. It must not be hurried, otherwise there may be loss of alcohol by imperfect condensation.

4. The condenser should, when in use, be kept filled with cold water. If the water used for condensation is above 80° F., ice should be added to cool it. The water should be kept circulating through the condenser during the distillation.

5. All measurements of the liquid should always be made as near as possible to the standard temperature, 60° F., otherwise sources of error are introduced into the determination.

6. The distillate should, after the necessary dilution with distilled water at 60° F., be thoroughly mixed, by closing the open end of the receiver with the palm of the hand and inverting the receiver and its contents four or five times, before attempting to make a reading with the alcoholometer.

7. Especial care must be taken that the distillate is brought back exactly to the volume and temperature at which the original measurement of the wine was made, and no attempt should be made to determine the specific gravity of the distillate until these conditions are exactly attained.

8. The hands must be perfectly clean. The least foreign matter, especially any of a fatty nature, if brought in contact with either the stem of the alcoholometer or thermometer, or with the surface of the alcohol itself, changes the indications of the alcoholometer and makes the readings too high. For this reason the alcoholometer and thermometer should be fingered as little as possible, and must be cleaned before they are used.

9. Readings of the thermometer and hydrometer must not be made until after the expiration of at least five minutes, to allow of the instruments attaining the same temperature as the surrounding liquid.

DETERMINATION OF THE SUGAR.

(1) The glass cylinder, previously thoroughly washed, is first rinsed with the sample, then filled to within a short distance of the top, and the temperature accurately noted. On the accuracy and care taken in making these readings depends the correctness of the determinations.

(2) The hydrometer (saccharometer) is then introduced into the liquid, and the same care and precaution observed as when the alcoholometer is used in ascertaining the strength of spirits. The saccharometer must be cleaned and dried before each determination. The reading of the scale of the saccharometer is made after the instrument has been in the liquid five minutes, by placing the eye on a level with the surface of the liquid, and not above or below it, and noting the first line on the stem corresponding to the general surface of the liquid, but not the line corresponding to the rising of the liquid on the stem by capillary attraction. The reading of the thermometer will also be taken at the same time.

(3) When the temperature is 60° F., the reading of the scale of the saccharometer will give at once the percentage of sugar by weight in the liquid corresponding to the specific gravity thus found. When the temperature, however, varies from this standard, the readings of the saccharometer must be corrected and brought to the standard temperature. For this purpose the officer will refer to Table I, and if the

observed temperature is below 60° F., he will subtract the decimal found in the table in the column headed by the degree in whole numbers nearest to the reading of the saccharometer on the line with the degree at the left of the table corresponding to the noted temperature, and if the observed temperature is above 60° F. he will add the decimal so found.

For example.

	Temperature of observation.	
	Below normal.	Above normal.
Noted temperature	56° F.	75° F.
Reading of saccharometer at noted temperature	13.4	12.6
Correction to be applied as found in Table I	—.11	+.55
Corrected reading of saccharometer at 60° F	13.29	13.15

(4) Fill the cylinder to the mark exactly with the liquid under examination, and note the temperature; transfer the contents of the cylinder to the evaporating dish (previously washed and dried), and rinse the cylinder with a little pure water; transfer the washings to the dish, and again rinse the cylinder with some more water, using as little as possible, as time will be lost in the evaporation of the same. Care must be taken not to spill or lose any of the measured sample or of the washings. Place the evaporating dish over the flame of the alcohol lamp, and let the contents evaporate slowly; the operation must not be hurried, as there is danger of the liquid boiling over if too great a heat is applied. Continue the process until at least one-half of the liquid has been driven off, then remove from the flame and let stand until cold. (The evaporation may be continued until more than one-half of the liquid has been expelled, but never to dryness nor to a thick sirup.) When cold, transfer back to the measuring cylinder and wash the dish with a little cold water; add the washings to the contents of the cylinder; repeat the washings once or twice. Then add enough water to bring the volume to within a quarter of an inch of the mark. The contents of the cylinder are now to be brought exactly to the temperature noted when the cylinder was filled with the original liquid, using the same method of cooling as mentioned in (2). When the liquid is at the noted temperature add sufficient water of the same temperature to bring the volume up to the mark, and no more.

[(5) The water to be used in these operations must be either distilled, condensed, rain water, or melted ice. Well and spring water must *not* be used. River water may be employed if no distilled, condensed, rain water, or melted ice be procurable.]

(6) Close the open end of the cylinder with the palm of the hand, and invert the cylinder and its contents slowly four or five times, in order to insure a thorough mixing of the different layers of liquid. Then insert the saccharometer, and make the reading as directed in (2), and the calculation as directed in (3). (This will give the percentage of sugar by weight in the liquid.)

(7) The first reading of the saccharometer (3) ascertains the specific gravity of the liquid at 60° F. compared with distilled water at the same temperature, and the second reading of the saccharometer (6) ascertains the density of the dealcoholized liquid at 60° F. Each degree of the saccharometer corresponds to a certain specific gravity. (Table I.)

For example.

(*a*) Temperature noted of *original liquid*, °F	75	
(*b*) Reading of saccharometer in *original liquid*		5.6
(*c*) Correction to be applied for temperature	add	.51
(*d*) Corrected reading of saccharometer at 60° F		6.11

AFTER DEALCOHOLIZATION.

(*e*) Temperature noted, °F	75
(*f*) Reading of saccharometer	12.6
(*g*) Correction to be applied for temperature	add .55
(*h*) Corrected reading of saccharometer at 60° F	13.15

Result of examination of liquid:

Per cent by weight of sugar (*h*)	13.15

TABLE I.—*Correction to be applied to the reading of Balling's saccharometer for different temperatures.*

	Temp., °F.	Degree Balling.																			
		1°	2°	3°	4°	5°	6°	7°	8°	9°	10°	11°	12°	13°	14°	15°	16°	17°	18°	19°	20°	21°
To be subtracted from the indicated degree.	50	.17	.17	.18	.19	.20	.20	.21	.21	.22	.22	.22	.23	.23	.24	.24	.24	.25	.25	.25	.26	.26
	51	.16	.16	.17	.18	.19	.19	.20	.20	.21	.21	.21	.22	.22	.23	.23	.23	.24	.24	.24	.24	.25
	52	.15	.15	.16	.17	.18	.18	.18	.19	.19	.19	.19	.19	.20	.20	.21	.21	.22	.22	.22	.22	.23
	53	.14	.14	.14	.15	.16	.16	.16	.17	.17	.18	.18	.18	.18	.19	.19	.19	.19	.20	.20	.20	.20
	54	.12	.12	.13	.14	.15	.15	.15	.16	.16	.16	.16	.17	.17	.17	.17	.17	.18	.18	.18	.18	.18
	55	.09	.09	.09	.10	.11	.11	.11	.12	.12	.12	.12	.13	.13	.13	.14	.14	.14	.14	.14	.14	.15
	56	.07	.07	.07	.08	.09	.09	.09	.09	.10	.10	.10	.10	.11	.11	.11	.11	.11	.11	.11	.11	.11
	57	.06	.06	.06	.07	.07	.07	.07	.07	.08	.08	.08	.08	.08	.09	.09	.09	.09	.10	.10	.10	.10
	58	.04	.04	.04	.05	.05	.05	.05	.05	.06	.06	.06	.06	.06	.07	.07	.07	.07	.07	.07	.07	.07
	59	.02	.02	.02	.02	.02	.02	.02	.02	.03	.03	.03	.03	.03	.03	.03	.03	.03	.03	.03	.03	.03
	60																					
To be added to the indicated degree.	61	.02	.02	.02	.03	.03	.03	.03	.03	.03	.03	.03	.03	.03	.03	.03	.03	.03	.03	.03	.03	.03
	62	.06	.06	.07	.07	.08	.08	.08	.08	.08	.08	.09	.09	.09	.09	.09	.09	.09	.09	.09	.09	.09
	63	.09	.10	.10	.11	.11	.11	.11	.11	.11	.11	.12	.12	.12	.12	.12	.12	.12	.12	.13	.12	.12
	64	.12	.12	.13	.13	.14	.14	.14	.14	.15	.15	.15	.15	.16	.16	.17	.17	.17	.17	.17	.17	.17
	65	.14	.15	.15	.16	.17	.17	.17	.17	.18	.18	.18	.19	.19	.19	.20	.20	.20	.20	.20	.20	.20
	66	.17	.17	.18	.19	.20	.20	.21	.21	.21	.22	.22	.23	.23	.23	.24	.24	.24	.24	.24	.24	.24
	67	.20	.21	.21	.22	.23	.23	.23	.24	.24	.25	.25	.25	.26	.26	.27	.27	.27	.27	.27	.27	.27
	68	.22	.23	.24	.25	.26	.26	.27	.28	.28	.29	.29	.30	.30	.31	.31	.31	.31	.31	.31	.31	.31
	69	.25	.26	.27	.28	.29	.29	.30	.31	.31	.32	.32	.33	.33	.34	.34	.34	.34	.34	.34	.34	.34
	70	.28	.29	.30	.31	.32	.32	.33	.34	.34	.35	.35	.36	.36	.37	.37	.37	.37	.38	.38	.38	.38
	71	.31	.32	.33	.34	.35	.35	.36	.37	.37	.38	.38	.39	.39	.40	.40	.40	.40	.40	.40	.40	.41
	72	.34	.35	.36	.37	.38	.39	.40	.41	.42	.42	.42	.43	.43	.43	.44	.44	.44	.44	.44	.44	.44
	73	.37	.38	.40	.41	.42	.43	.44	.44	.45	.46	.46	.46	.46	.47	.47	.47	.47	.47	.47	.47	.47
	74	.40	.42	.43	.45	.46	.47	.48	.48	.49	.50	.50	.50	.51	.51	.51	.51	.51	.52	.52	.52	.52
	75	.44	.46	.47	.49	.50	.51	.52	.52	.53	.54	.54	.54	.55	.55	.56	.56	.56	.57	.57	.58	.58
	76	.47	.48	.50	.52	.53	.54	.55	.55	.56	.57	.57	.58	.58	.59	.60	.60	.61	.61	.61	.62	.62
	77	.50	.52	.54	.56	.57	.58	.59	.60	.60	.61	.61	.62	.62	.63	.63	.63	.64	.64	.64	.65	.65
	78	.53	.54	.56	.58	.60	.61	.62	.62	.63	.64	.64	.65	.65	.66	.66	.66	.67	.67	.68	.68	.68
	79	.57	.58	.60	.62	.64	.65	.66	.66	.67	.68	.68	.69	.69	.70	.70	.70	.71	.71	.71	.72	.72
	80	.60	.62	.64	.66	.68	.69	.70	.70	.71	.72	.72	.72	.73	.73	.74	.74	.75	.75	.75	.76	.76

To be added to the indicated degree.																						
81	.63	.65	.67	.69	.71	.72	.73	.73	.74	.75	.75	.76	.76	.77	.78	.78	.79	.79	.79	.79	79
82	.67	.69	.71	.73	.76	.76	.77	.77	.78	.78	.79	.80	.80	.81	.82	.82	.83	.83	.84	.84	.84
83	.72	.74	.76	.78	.81	.81	.81	.82	.82	.82	.83	.84	.85	.86	.87	.87	.87	.87	.87	.87	.88
84	.77	.79	.81	.83	.86	.86	.86	.86	.87	.87	.88	.89	.90	.91	.92	.92	.93	.93	.93	.93	.94
85	.83	.85	.87	.89	.91	.91	.92	.92	.93	.93	.94	.95	.96	.97	.98	.98	.98	.99	.99	.99	.99
86	.88	.90	.92	.94	.96	.96	.97	.98	.98	.99	1.00	1.01	1.02	1.02	1.03	1.03	1.04	1.04	1.04	1.05	1.05
87	.94	.96	.98	.99	1.01	1.02	1.03	1.03	1.04	1.05	1.05	1.06	1.07	1.07	1.08	1.08	1.09	1.10	1.10	1.11	1.11
88	.99	1.01	1.03	1.04	1.06	1.07	1.08	1.09	1.10	1.11	1.11	1.12	1.12	1.13	1.13	1.14	1.15	1.15	1.16	1.17	1.17
89	1.05	1.07	1.09	1.10	1.11	1.15	1.13	1.14	1.15	1.16	1.16	1.17	1.17	1.18	1.18	1.19	1.21	1.21	1.22	1.23	1.23
90	1.11	1.13	1.15	1.16	1.17	1.18	1.19	1.20	1.21	1.22	1.22	1.23	1.23	1.24	1.24	1.25	1.27	1.28	1.29	1.30	1.30
91	1.15	1.17	1.19	1.20	1.21	1.22	1.23	1.24	1.26	1.27	1.27	1.27	1.28	1.28	1.29	1.30	1.32	1.33	1.34	1.35	1.36
92	1.20	1.22	1.24	1.25	1.26	1.27	1.28	1.30	1.31	1.32	1.32	1.33	1.33	1.34	1.34	1.35	1.37	1.38	1.39	1.40	1.41
93	1.24	1.26	1.28	1.29	1.31	1.32	1.33	1.34	1.36	1.37	1.37	1.38	1.39	1.39	1.40	1.40	1.42	1.43	1.44	1.44	1.45
94	1.29	1.31	1.33	1.35	1.36	1.37	1.39	1.40	1.41	1.42	1.42	1.43	1.44	1.44	1.45	1.45	1.47	1.47	1.48	1.49	1.50
95	1.33	1.35	1.37	1.39	1.41	1.42	1.44	1.45	1.46	1.47	1.47	1.48	1.49	1.49	1.50	1.50	1.51	1.52	1.53	1.54	1.54
96	1.38	1.40	1.42	1.44	1.46	1.47	1.49	1.50	1.51	1.52	1.52	1.53	1.54	1.54	1.55	1.55	1.56	1.57	1.57	1.58	1.58
97	1.42	1.44	1.46	1.48	1.51	1.52	1.54	1.55	1.56	1.57	1.57	1.58	1.59	1.59	1.60	1.60	1.62	1.62	1.63	1.63	1.64
98	1.47	1.49	1.51	1.54	1.56	1.57	1.58	1.59	1.60	1.62	1.63	1.63	1.64	1.64	1.65	1.65	1.66	1.67	1.67	1.68	1.68
99	1.51	1.53	1.55	1.58	1.61	1.62	1.63	1.64	1.66	1.67	1.68	1.68	1.69	1.70	1.71	1.71	1.72	1.73	1.73	1.73	1.74
100	1.55	1.57	1.59	1.62	1.66	1.67	1.68	1.69	1.72	1.72	1.73	1.73	1.74	1.76	1.77	1.77	1.78	1.79	1.79	1.79	1.80

N. B.—The correction for indications between 21° and 30° Balling and 50° and 70° F. temperature may be made by adding 0.005 to each correction given in the above Table, column 21°, for each increase in degree Balling. For instance, 25° Balling 70° F., correction to be added to column 21° and 70°. 0.02+0.38=0.40.

Table II.

Specific gravity at 60° F., corresponding to readings of Balling's saccharometer.

[W. Schultze, Dingler's Polytechnisches Journal, 1878, 230, p. 421.]

Balling.	Specific gravity.	Balling.	Specific gravity.	Balling.	Specific gravity.	Balling.	Specific gravity.	Balling.	Specific gravity.
0.00	1.0000	1.31	1.0050	2.58	1.0100	3.87	1.0150	5.20	1.0200
0.03	1.0001	1.34	1.0051	2.61	1.0101	3.90	1.0151	5.23	1.0201
0.05	1.0002	1.36	1.0052	2.64	1.0102	3.92	1.0152	5.25	1.0202
0.08	1.0003	1.39	1.0053	2.66	1.0103	3.95	1.0153	5.28	1.0203
0.10	1.0004	1.41	1.0054	2.69	1.0104	3.97	1.0154	5.30	1.0204
0.13	1.0005	1.44	1.0055	2.71	1.0105	4.00	1.0155	5.33	1.0205
0.16	1.0006	1.46	1.0056	2.74	1.0106	4.03	1.0156	5.35	1.0206
0.18	1.0007	1.49	1.0057	2.76	1.0107	4.05	1.0157	5.38	1.0207
0.21	1.0008	1.51	1.0058	2.79	1.0108	4.08	1.0158	5.40	1.0208
0.24	1.0009	1.54	1.0059	2.82	1.0109	4.10	1.0159	5.43	1.0209
0.26	1.0010	1.56	1.0060	2.84	1.0110	4.13	1.0160	5.45	1.0210
0.29	1.0011	1.59	1.0061	2.87	1.0111	4.16	1.0161	5.48	1.0211
0.31	1.0012	1.62	1.0062	2.89	1.0112	4.18	1.0162	5.50	1.0212
0.34	1.0013	1.64	1.0063	2.92	1.0113	4.21	1.0163	5.53	1.0213
0.37	1.0014	1.67	1.0064	2.94	1.0114	4.23	1.0164	5.55	1.0214
0.39	1.0015	1.69	1.0065	2.97	1.0115	4.26	1.0165	5.57	1.0215
0.42	1.0016	1.72	1.0066	2.99	1.0116	4.28	1.0166	5.60	1.0216
0.45	1.0017	1.74	1.0067	3.02	1.0117	4.31	1.0167	5.62	1.0217
0.47	1.0018	1.77	1.0068	3.05	1.0118	4.34	1.0168	5.65	1.0218
0.50	1.0019	1.79	1.0069	3.07	1.0119	4.36	1.0169	5.67	1.0219
0.52	1.0020	1.82	1.0070	3.10	1.0120	4.39	1.0170	5.70	1.0220
0.55	1.0021	1.84	1.0071	3.12	1.0121	4.42	1.0171	5.72	1.0221
0.58	1.0022	1.87	1.0072	3.15	1.0122	4.44	1.0172	5.75	1.0222
0.60	1.0023	1.90	1.0073	3.17	1.0123	4.47	1.0173	5.77	1.0223
0.63	1.0024	1.92	1.0074	3.20	1.0124	4.50	1.0174	5.80	1.0224
0.66	1.0025	1.95	1.0075	3.23	1.0125	4.53	1.0175	5.82	1.0225
0.68	1.0026	1.97	1.0076	3.25	1.0126	4.55	1.0176	5.84	1.0226
0.71	1.0027	2.00	1.0077	3.28	1.0127	4.58	1.0177	5.87	1.0227
0.73	1.0028	2.02	1.0078	3.30	1.0128	4.61	1.0178	5.89	1.0228
0.76	1.0029	2.05	1.0079	3.33	1.0129	4.63	1.0179	5.92	1.0229
0.79	1.0030	2.07	1.0080	3.35	1.0130	4.66	1.0180	5.94	1.0230
0.81	1.0031	2.10	1.0081	3.38	1.0131	4.69	1.0181	5.97	1.0231
0.84	1.0032	2.12	1.0082	3.41	1.0132	4.71	1.0182	5.99	1.0232
0.87	1.0033	2.15	1.0083	3.43	1.0133	4.74	1.0183	6.02	1.0233
0.89	1.0034	2.17	1.0084	3.46	1.0134	4.77	1.0184	6.04	1.0234
0.92	1.0035	2.20	1.0085	3.48	1.0135	4.79	1.0185	6.07	1.0235
0.94	1.0036	2.23	1.0086	3.51	1.0136	4.82	1.0186	6.09	1.0236
0.97	1.0037	2.25	1.0087	3.54	1.0137	4.85	1.0187	6.11	1.0237
1.00	1.0038	2.28	1.0088	3.56	1.0138	4.88	1.0188	6.14	1.0238
1.02	1.0039	2.30	1.0089	3.59	1.0139	4.90	1.0189	6.16	1.0239
1.05	1.0040	2.33	1.0090	3.61	1.0140	4.93	1.0190	6.19	1.0240
1.08	1.0041	2.35	1.0091	3.64	1.0141	4.96	1.0191	6.21	1.0241
1.10	1.0042	2.38	1.0092	3.66	1.0142	4.98	1.0192	6.24	1.0242
1.13	1.0043	2.41	1.0093	3.69	1.0143	5.01	1.0193	6.26	1.0243
1.15	1.0044	2.43	1.0094	3.72	1.0144	5.04	1.0194	6.29	1.0244
1.18	1.0045	2.46	1.0095	3.74	1.0145	5.06	1.0195	6.31	1.0245
1.21	1.0046	2.48	1.0096	3.77	1.0146	5.09	1.0196	6.34	1.0246
1.23	1.0047	2.51	1.0097	3.79	1.0147	5.12	1.0197	6.36	1.0247
1.26	1.0048	2.53	1.0098	3.82	1.0148	5.15	1.0198	6.39	1.0248
1.29	1.0049	2.56	1.0099	3.85	1.0149	5.17	1.0199	6.41	1.0249

TABLE II—Continued.

Specific gravity at 60° F., corresponding to readings of Balling's saccharometer.

[W. Schultze, Dingler's Polytechnisches Journal, 1878, 230, p. 421.]

Balling.	Specific gravity.	Balling.	Specific gravity.	Balling.	Specific gravity.	Balling.	Specific gravity.	Balling.	Specific gravity.
6.44	1.0250	7.71	1.0300	8.94	1.0350	10.16	1.0400	11.40	1.0450
6.47	1.0251	7.73	1.0301	8.97	1.0351	10.18	1.0401	11.43	1.0451
6.50	1.0252	7.75	1.0302	**8.99**	**1.0352**	10.20	1.0402	11.45	1.0452
6.52	1.0253	7.77	1.0303	9.02	1.0353	10.23	1.0403	11.48	1.0453
6.55	1.0254	7.80	1.0304	9.05	1.0354	10.25	1.0404	11.50	1.0454
6.58	1.0255	7.82	1.0305	9.07	1.0355	10.27	1.0405	11.53	1.0455
6.61	1.0256	7.84	1.0306	9.10	1.0356	10.30	1.0406	11.55	1.0456
6.63	1.0257	7.86	1.0307	9.13	1.0357	10.32	1.0407	11.57	1.0457
6.66	1.0258	7.89	1.0308	9.15	1.0358	10.35	1.0408	11.60	1.0458
6.69	1.0259	7.91	1.0309	9.18	1.0359	10.37	1.0409	11.62	1.0459
6.71	1.0260	7.93	1.0310	9.21	1.0360	10.40	1.0410	11.65	1.0460
6.74	1.0261	7.95	1.0311	9.24	1.0361	10.42	1.0411	11.67	1.0461
6.77	1.0262	7.98	1.0312	9.26	1.0362	10.45	1.0412	11.70	1.0462
6.80	1.0263	**8.00**	**1.0313**	9.29	1.0363	10.47	1.0413	11.72	1.0463
6.82	1.0264	8.02	1.0314	9.31	1.0364	10.50	1.0414	11.75	1.0464
6.85	1.0265	8.04	1.0315	9.34	1.0365	10.52	1.0415	11.77	1.0465
6.88	1.0266	8.07	1.0316	9.36	1.0366	10.55	1.0416	11.79	1.0466
6.91	1.0267	8.09	1.0317	9.38	1.0367	10.57	1.0417	11.82	1.0467
6.93	1.0268	8.11	1.0318	9.41	1.0368	10.60	1.0418	11.84	1.0468
6.96	1.0269	8.13	1.0319	9.43	1.0369	10.62	1.0419	11.87	1.0469
6.99	**1.0270**	8.16	1.0320	9.45	1.0370	10.65	1.0420	11.89	1.0470
7.01	1.0271	8.18	1.0321	9.48	1.0371	10.67	1.0421	11.92	1.0471
7.04	1.0272	8.20	1.0322	9.50	1.0372	10.70	1.0422	11.94	1.0472
7.07	1.0273	8.22	1.0323	9.52	1.0373	10.72	1.0423	11.97	1.0473
7.10	1.0274	8.25	1.0324	9.55	1.0374	10.75	1.0424	11.99	1.0474
7.12	1.0275	8.27	1.0325	9.57	1.0375	10.77	1.0425	**12.01**	**1.0475**
7.15	1.0276	8.29	1.0326	9.59	1.0376	10.80	1.0426	12.04	1.0476
7.18	1.0277	8.32	1.0327	9.62	1.0377	10.82	1.0427	12.06	1.0477
7.21	1.0278	8.34	1.0328	9.64	1.0378	10.85	1.0428	12.09	1.0478
7.23	1.0279	8.37	1.0329	9.66	1.0379	10.88	1.0429	12.11	1.0479
7.26	1.0280	8.40	1.0330	9.69	1.0380	10.90	1.0430	12.14	1.0480
7.28	1.0281	8.43	1.0331	9.71	1.0381	10.93	1.0431	12.16	1.0481
7.30	1.0282	8.45	1.0332	9.73	1.0382	10.95	1.0432	12.19	1.0482
7.33	1.0283	8.48	1.0333	9.76	1.0383	10.98	1.0433	12.21	1.0483
7.35	1.0284	8.51	1.0334	9.78	1.0384	**11.00**	**1.0434**	12.23	1.0484
7.37	1.0285	8.53	1.0335	9.81	1.0385	11.03	1.0435	12.26	1.0485
7.39	1.0286	8.56	1.0336	9.83	1.0386	11.05	1.0436	12.28	1.0486
7.42	1.0287	8.59	1.0337	9.85	1.0387	11.08	1.0437	12.31	1.0487
7.44	1.0288	8.61	1.0338	9.88	1.0388	11.10	1.0438	12.33	1.0488
7.46	1.0289	8.64	1.0339	9.90	1.0389	11.13	1.0439	12.36	1.0489
7.48	1.0290	8.67	1.0340	9.92	1.0390	11.15	1.0440	12.38	1.0490
7.51	1.0291	8.70	1.0341	9.95	1.0391	11.18	1.0441	12.41	1.0491
7.53	1.0292	8.72	1.0342	9.97	1.0392	11.20	1.0442	12.43	1.0492
7.55	1.0293	8.75	1.0343	**9.99**	**1.0393**	11.23	1.0443	12.45	1.0493
7.57	1.0294	8.78	1.0344	10.02	1.0394	11.25	1.0444	12.48	1.0494
7.60	1.0295	8.80	1.0345	10.04	1.0395	11.28	1.0445	12.50	1.0495
7.62	1.0296	8.83	1.0346	10.06	1.0396	11.30	1.0446	12.53	1.0496
7.64	1.0297	8.86	1.0347	10.09	1.0397	11.33	1.0447	12.55	1.0497
7.66	1.0298	8.88	1.0348	10.11	1.0398	11.35	1.0448	12.58	1.0498
7.69	**1.0299**	**8.91**	**1.0349**	**10.13**	1.0399	11.38	1.0449	**12.60**	1.0499

TABLE II—Continued.

Specific gravity at 60° F., corresponding to readings of Balling's saccharometer.

[W. Schultze. Dingler's Polytechnisches Journal, 1878, 230, p. 421.]

Balling.	Specific gravity.	Balling.	Specific gravity.	Balling.	Specific gravity.	Balling.	Specific gravity.	Balling.	Specific gravity.
12.63	1.0500	13.86	1.0550	15.14	1.0600	16.25	1.0650	17.48	1.0700
12.65	1.0501	13.88	1.0551	15.16	1.0601	16.27	1.0651	17.50	1.0701
12.67	1.0502	13.91	1.0552	15.18	1.0602	16.30	1.0652	17.52	1.0702
12.70	1.0503	13.93	1.0553	15.20	1.0603	16.32	1.0653	17.54	1.0703
12.72	1.0504	13.96	1.0554	15.23	1.0604	16.35	1.0654	17.57	1.0704
12.75	1.0505	13.98	1.0555	15.25	1.0605	16.37	1.0655	17.59	1.0705
12.77	1.0506	**14.01**	**1.0556**	15.27	1.0606	16.40	1.0656	17.61	1.0706
12.80	1.0507	14.03	1.0557	15.29	1.0607	16.42	1.0657	17.63	1.0707
12.82	1.0508	14.06	1.0558	15.31	1.0608	16.45	1.0658	17.66	1.0708
12.85	1.0509	14.08	1.0559	15.34	1.0609	16.47	1.0659	17.68	1.0709
12.87	1.0510	14.11	1.0560	15.36	1.0610	16.50	1.0660	17.70	1.0710
12.90	1.0511	14.13	1.0561	15.38	1.0611	16.52	1.0661	17.72	1.0711
12.92	1.0512	14.16	1.0562	15.40	1.0612	16.54	1.0662	17.75	1.0712
12.94	1.0513	14.18	1.0563	15.43	1.0613	16.57	1.0663	17.77	1.0713
12.97	1.0514	14.21	1.0564	15.45	1.0614	16.59	1.0664	17.79	1.0714
12.99	**1.0515**	14.23	1.0565	15.47	1.0615	16.62	1.0665	17.81	1.0715
13.02	1.0516	14.26	1.0566	15.49	1.0616	16.64	1.0666	17.84	1.0716
13.04	1.0517	14.28	1.0567	15.52	1.0617	16.67	1.0667	17.86	1.0717
13.07	1.0518	14.31	1.0568	15.54	1.0618	16.69	1.0668	17.88	1.0718
13.09	1.0519	14.33	1.0569	15.56	1.0619	16.72	1.0669	17.90	1.0719
13.12	1.0520	14.36	1.0570	15.58	1.0620	16.74	1.0670	17.93	1.0720
13.14	1.0521	14.38	1.0571	15.60	1.0621	16.76	1.0671	17.95	1.0721
13.16	1.0522	14.41	1.0572	15.63	1.0622	16.79	1.0672	17.97	1.0722
13.19	1.0523	14.44	1.0573	15.65	1.0623	16.81	1.0673	**17.99**	**1.0723**
13.21	1.0524	14.46	1.0574	15.67	1.0624	16.84	1.0674	18.02	1.0724
13.24	1.0525	14.49	1.0575	15.69	1.0625	16.86	1.0675	18.04	1.0725
13.26	1.0526	14.52	1.0576	15.72	1.0626	16.89	1.0676	18.06	1.0726
13.29	1.0527	14.54	1.0577	15.74	1.0627	16.91	1.0677	18.08	1.0727
13.31	1.0528	14.57	1.0578	15.76	1.0628	16.94	1.0678	18.11	1.0728
13.34	1.0529	14.59	1.0579	15.78	1.0629	16.96	1.0679	18.13	1.0729
13.36	1.0530	14.62	1.0580	15.80	1.0630	**16.99**	**1.0680**	18.15	1.0730
13.38	1.0531	14.65	1.0581	15.83	1.0631	17.01	1.0681	18.17	1.0731
13.41	1.0532	14.67	1.0582	15.85	1.0632	17.03	1.0682	18.20	1.0732
13.43	1.0533	14.70	1.0583	15.87	1.0633	17.06	1.0683	18.22	1.0733
13.46	1.0534	14.73	1.0584	15.89	1.0634	17.08	1.0684	18.24	1.0734
13.48	1.0535	14.75	1.0585	15.92	1.0635	17.11	1.0685	18.26	1.0735
13.51	1.0536	14.78	1.0586	15.94	1.0636	17.13	1.0686	18.29	1.0736
13.53	1.0537	14.81	1.0587	15.96	1.0637	17.16	1.0687	18.31	1.0737
13.56	1.0538	14.83	1.0588	15.98	1.0638	17.18	1.0688	18.33	1.0738
13.58	1.0539	14.86	1.0589	**16.01**	**1.0639**	17.21	1.0689	18.35	1.0739
13.61	1.0540	14.89	1.0590	16.03	1.6040	17.23	1.0690	18.38	1.0740
13.63	1.0541	14.91	1.0591	16.05	1.6041	17.25	1.0691	18.40	1.0741
13.66	1.0542	14.94	1.0592	16.07	1.6042	17.28	1.0692	18.42	1.0742
13.68	1.0543	14.96	1.0593	16.09	1.6043	17.30	1.0693	18.44	1.0743
13.71	1.0544	**14.99**	**1.0594**	16.12	1.6044	17.33	1.0694	18.47	1.0744
13.73	1.0545	15.02	1.0595	16.14	1.0645	17.35	1.0695	18.49	1.0745
13.76	1.0546	15.04	1.0596	16.16	1.0646	17.38	1.0696	18.51	1.0746
13.78	1.0547	15.07	1.0597	16.18	1.0647	17.40	1.0697	18.53	1.0747
13.81	1.0548	15.09	1.0598	16.21	1.0648	17.43	1.0698	18.55	1.0748
13.83	1.0549	15.11	1.0599	16.23	1.0649	17.45	1.0699	18.57	1.0749

TABLE II—Continued.

Specific gravity at 60° F., corresponding to readings of Balling's saccharometer.

[W. Schultze. Dingler's Polytechnisches Journal, 1878, 230, p. 421.]

Balling.	Specific gravity.	Balling.	Specific gravity.	Balling.	Specific gravity.	Balling.	Specific gravity.	Balling.	Specific gravity.
18.59	1.0750	19.33	1.0785	20.6	1.0841	24.1	1.0999	27.6	1.1161
18.62	1.0751	19.36	1.0786	20.7	1.0845	24.2	1.1003	27.7	1.1166
18.64	1.0752	19.38	1.0787	20.8	1.0850	24.3	1.1008	27.8	1.1170
18.66	1.0753	19.40	1.0788	20.9	1.0854	24.4	1.1012	27.9	1.1175
18.68	1.0754	19.42	1.0789	**21.0**	**1.0859**	24.5	1.1017	**28.0**	**1.1180**
18.70	1.0755	19.44	1.0790	21.1	1.0863	24.6	1.1022	28.1	1.1184
18.72	1.0756	19.46	1.0791	21.2	1.0867	24.7	1.1026	28.2	1.1189
18.74	1.0757	19.49	1.0792	21.3	1.0872	24.8	1.1031	28.3	1.1194
18.76	1.0758	19.51	1.0793	21.4	1.0876	24.9	1.1035	28.4	1.1198
18.78	1.0759	19.53	1.0794	21.5	1.0881	**25.0**	**1.1040**	28.5	1.1203
18.81	1.0760	19.56	1.0795	21.6	1.0885	25.1	1.1045	28.6	1.1208
18.83	1.0761	19.58	1.0796	21.7	1.0890	25.2	1.1049	28.7	1.1213
18.85	1.0762	19.60	1.0797	21.8	1.0894	25.3	1.1054	28.8	1.1217
18.87	1.0763	19.63	1.0798	21.9	1.0899	25.4	1.1058	28.9	1.1222
18.89	1.0764	19.65	1.0799	**22.0**	**1.0903**	25.5	1.1063	**29.0**	**1.1227**
18.91	1.0765	19.67	1.0800	22.1	1.0908	25.6	1.1068	29.1	1.1232
18.93	1.0766	19.70	1.0801	22.2	1.0912	25.7	1.1072	29.2	1.1236
18.95	1.0767	19.72	1.0802	22.3	1.0917	25.8	1.1077	29.3	1.1241
18.97	1.0768	19.74	1.0803	22.4	1.0921	25.9	1.1082	29.4	1.1246
19.00	**1.0769**	19.77	1.0804	22.5	1.0926	**26.0**	**1.1086**	29.5	1.1251
19.02	1.0770	19.79	1.0805	22.6	1.0931	26.1	1.1091	29.6	1.1255
19.04	1.0771	19.81	1.0806	22.7	1.0935	26.2	1.1096	29.7	1.1260
19.06	1.0772	19.84	1.0807	22.8	1.0940	26.3	1.1100	29.8	1.1265
19.08	1.0773	19.86	1.0808	22.9	1.0944	26.4	1.1105	29.9	1.1270
19.10	1.0774	19.88	1.0809	**23.0**	**1.0949**	26.5	1.1109	**30.0**	**1.1274**
19.12	1.0775	19.91	1.0810	23.1	1.0953	26.6	1.1114		
19.14	1.0776	19.93	1.0811	23.2	1.0958	26.7	1.1119		
19.17	1.0777	19.96	1.0812	23.3	1.0962	26.8	1.1123		
19.19	1.0778	19.98	1.0813	23.4	1.0967	26.9	1.1128		
19.21	1.0779	**20.0**	**1.0814**	23.5	1.0971	**27.0**	**1.1133**		
19.23	1.0780	20.1	1.0818	23.6	1.0976	27.1	1.1137		
19.25	1.0781	20.2	1.0823	23.7	1.0980	27.2	1.1142		
19.27	1.0782	20.3	1.0827	23.8	1.0985	27.3	1.1147		
19.29	1.0783	20.4	1.0832	23.9	1.0990	27.4	1.1151		
19.31	1.0784	20.5	1.0836	**24.0**	**1.0994**	27.5	1.1165		

TABLE III.—*Specific gravity of alcohol by volume at 60° F.*

[Recalculated from Gilpin's, Drinkwater's, Squibb's, etc., determination, by EDGAR RICHARDS.]

Specific gravity at 60° F.	Per cent alcohol by volume.	Per cent alcohol by weight.	Specific gravity at 60° F.	Per cent alcohol by volume.	Per cent alcohol by weight.	Specific gravity at 60° F.	Per cent alcohol by volume.	Per cent alcohol by weight	Specific gravity at 60° F.	Per cent alcohol by volume.	Per cent alcohol by weight
1.00000	**0.00**	**0.00**	**.99629**	**2.50**	**1.99**	**.99281**	**5.00**	**4.00**	**.98959**	**7.50**	**6.02**
0.99992	.05	.04	622	.55	2.03	274	.05	.04	953	.55	.06
984	.10	.08	615	.60	.07	268	.10	.08	947	.60	.10
976	.15	.12	607	.65	.11	261	.15	.12	940	.65	.14
968	.20	.16	600	.70	.15	255	.20	.16	934	.70	.18
961	.25	.20	593	.75	.19	248	.25	.20	928	.75	.22
953	.30	.24	586	.80	.23	241	.30	.24	922	.80	.26
945	.35	.28	579	.85	.27	235	.35	.28	916	.85	.30
937	.40	.32	571	.90	.31	228	.40	.32	909	.90	.34
930	.45	.36	564	.95	.35	222	.45	.36	903	.95	.38
.99923	**0.50**	**0.40**	**.99557**	**3.00**	**2.39**	**.99215**	**5.50**	**4.40**	**.98897**	**8.00**	**6.42**
915	.55	.44	550	.05	.43	208	.55	.44	891	.05	.46
907	.60	.48	543	.10	.47	202	.60	.48	885	.10	.50
900	.65	.52	336	.15	.51	195	.65	.52	879	.15	.54
892	.70	.56	529	.20	.55	189	.70	.56	873	.20	.58
884	.75	.60	522	.25	.59	182	.75	.60	867	.25	.62
877	.70	.64	515	.30	.64	175	.80	.64	861	.30	.67
869	.85	.67	508	.35	.68	169	.85	.68	855	.35	.71
861	.90	.71	501	.40	.72	162	.90	.72	849	.40	.75
854	.95	.75	494	.45	.76	156	.95	.76	843	.45	.79
.99849	**1.00**	**0.79**	**.99487**	**3.50**	**2.80**	**.99149**	**6.00**	**4.80**	**.98837**	**8.50**	**6.83**
842	.05	.83	480	.55	.84	143	.05	.84	831	.55	.87
834	.10	.87	473	.60	.88	136	.10	.88	825	.60	.91
827	.15	.91	466	.65	.92	130	.15	.92	819	.65	.95
819	.20	.95	459	.70	.96	123	.20	.96	813	.70	.99
812	.25	.99	452	.75	3.00	117	.25	5.00	807	.75	7.03
805	.30	1.03	445	.80	.04	111	.30	.05	801	.80	.07
797	.35	.07	438	.85	.08	104	.35	.09	795	.85	.11
790	.40	.11	431	.90	.12	098	.40	.13	789	.90	.15
782	.45	.15	424	.95	.16	091	.45	.17	783	.95	.19
.99775	**1.50**	**1.19**	**.99417**	**4.00**	**3.20**	**.99085**	**6.50**	**5.21**	**.98777**	**9.00**	**7.23**
768	.55	.23	410	.05	.24	079	.55	.25	771	.05	.27
760	.60	.27	403	.10	.28	072	.60	.29	765	.10	.31
753	.65	.31	397	.15	.32	066	.65	.33	759	.15	.35
745	.70	.35	390	.20	.36	059	.70	.37	754	.20	.39
738	.75	.39	383	.25	.40	053	.75	.41	748	.25	.43
731	.80	.43	376	.30	.44	047	.80	.45	742	.30	.48
723	.85	.47	369	.35	.48	040	.85	.49	736	.35	.52
716	.90	.51	363	.40	.52	034	.90	.53	730	.40	.56
708	.95	.55	356	.45	.56	027	.95	.57	724	.45	.60
.99701	**2.00**	**1.59**	**.99349**	**4.50**	**3.60**	**.99021**	**7.00**	**5.61**	**.98719**	**9.50**	**7.64**
694	.05	.63	342	.55	.64	015	.05	.65	713	.55	.68
687	.10	.67	335	.60	.68	009	.10	.69	707	.60	.72
679	.15	.71	329	.65	.72	002	.15	.73	701	.65	.76
672	.20	.75	322	.70	.76	.98996	.20	.77	695	.70	.80
665	.25	.79	315	.75	.80	990	.25	.81	689	.75	.84
658	.30	.83	308	.80	.84	984	.30	.86	683	.80	.88
651	.35	.87	301	.85	.88	978	.35	.90	678	.85	.92
643	.30	.91	295	.90	.92	971	.40	.94	672	.90	.96
636	.45	.95	288	.95	.96	965	.45	.98	666	.95	8.00

TABLE III.—*Specific gravity of alcohol by volume at* 60° *F.*—Continued.

Specific gravity at 60° F.	Per cent alcohol by volume.	Per cent alcohol by weight.	Specific gravity at 60° F.	Per cent alcohol by volume.	Per cent alcohol by weight.	Specific gravity at 60° F.	Per cent alcohol by volume.	Per cent alcohol by weight.	Specific gravity at 60° F.	Per cent alcohol by volume.	Per cent alcohol by weight.
.98660	10.00	8.04	.98381	12.50	10.08	.98114	15.00	12.13	.97859	17.50	14.19
654	.05	.08	375	.55	.12	108	.05	.17	853	.55	.23
649	.10	.12	370	.60	.16	104	.10	.21	848	.60	.27
643	.15	.16	364	.65	.20	099	.15	.25	843	.65	.31
637	.20	.20	359	.70	.24	093	.20	.29	838	.70	.35
632	.25	.24	353	.75	.28	088	.25	.33	833	.75	.40
626	.30	.29	348	.80	.33	083	.30	.38	828	.80	.44
620	.35	.33	342	.85	.37	078	.35	.42	823	.85	.48
614	.40	.37	337	.90	.41	073	.40	.46	818	.90	.52
609	.45	.41	331	.95	.45	068	.45	.50	813	.95	.56
.98603	10.50	8.45	.98326	13.00	10.49	.98063	15.50	12.54	.97808	18.00	14.60
597	.55	.49	321	.05	.53	057	.55	.58	803	.05	.64
592	.60	.53	315	.10	.57	052	.60	.62	798	.10	.68
586	.65	.57	310	.15	.61	047	.65	.66	793	.15	.73
580	.70	.61	305	.20	.65	042	.70	.70	788	.20	.77
575	.75	.65	299	.25	.69	037	.75	.75	783	.25	.81
569	.80	.70	294	.30	.74	032	.80	.79	778	.30	.85
563	.85	.74	289	.35	.78	026	.85	.83	773	.35	.89
557	.90	.78	283	.40	.82	021	.90	.87	768	.40	.94
552	.95	.82	278	.45	.86	016	.95	.91	763	.45	.98
.98546	11.00	8.86	.98273	13.50	10.90	.98011	16.00	12.95	.97758	18.50	15.02
540	.05	.90	267	.55	.94	005	.05	.99	753	.55	.06
535	.10	.94	262	.60	.98	001	.10	13.03	748	.60	.10
529	.15	.98	256	.65	11.02	.97996	.15	.08	743	.65	.14
524	.20	9.02	251	.70	.06	991	.20	.12	738	.70	.18
518	.25	.07	246	.75	.11	986	.25	.16	733	.75	.22
513	.30	.11	240	.80	.15	980	.30	.20	728	.80	.27
507	.35	.15	235	.85	.19	975	.35	.24	723	.85	.31
502	.40	.19	230	.90	.23	970	.40	.29	718	.90	.38
496	.45	.23	224	.95	.27	965	.45	.33	713	.95	.39
.98491	11.50	9.27	98.219	14.00	11.31	.97960	16.50	13.37	.97708	19.00	15.43
485	.55	.31	214	.05	.35	955	.55	.41	703	.05	.47
479	.60	.35	209	.10	.39	950	.60	.45	698	.10	.51
474	.65	.39	203	.15	.43	945	.65	.49	693	.15	.55
468	.70	.43	198	.20	.47	940	.70	.53	688	.20	.59
463	.75	.47	193	.25	.52	935	.75	.57	683	.25	.63
457	.80	.51	188	.30	.56	929	.80	.62	678	.30	.68
452	.85	.55	182	.35	.60	924	.85	.66	673	.35	.72
446	.90	.59	177	.40	.64	919	.90	.70	668	.40	.76
441	.95	.63	172	.45	.68	914	.95	.74	663	.45	80
.98435	12.00	9.67	.98167	14.50	11.72	.97909	17.00	13.78	.97658	19.50	15.84
430	.05	.71	161	.55	.76	904	.05	.82	653	.55	.88
424	.10	.75	156	.60	.80	899	.10	.86	648	.60	.93
419	.15	.79	151	.65	.84	894	.15	.90	643	.65	.97
413	.20	.83	146	.70	.88	889	.20	.94	638	.70	16.01
408	.25	.87	140	.75	.93	884	.25	.98	633	.75	.05
402	.30	.92	135	.80	.97	879	.30	14.03	628	.80	.09
397	.35	.96	130	.85	12.05	874	.35	.07	623	.85	.14
391	.40	10.00	125	.90	.05	869	.40	.11	618	.90	.18
386	.45	.04	119	.95	.09	864	.45	.15	613	.95	.22

TABLE III.—*Specific gravity of alcohol by volume at* 60° *F.*—Continued.

Specific gravity at $\frac{60°}{60°}$ F.	Per cent alcohol by volume.	Per cent alcohol by weight.	Specific gravity at $\frac{60°}{60°}$ F.	Per cent alcohol by volume.	Per cent alcohol by weight	Specific gravity at $\frac{60°}{60°}$ F.	Per cent alcohol by volume.	Per cent alcohol by weight.	Specific gravity at $\frac{60°}{60°}$ F.	Per cent alcohol by volume.	Per cent alcohol by weight.
.97608	20.00	16.26	.97355	22.50	18.34	.97097	25.00	20.43	.96828	27.50	22.54
603	.05	.30	350	.55	.38	092	.05	.47	822	.55	.58
598	.10	.34	345	.60	.42	086	.10	.51	816	.60	.62
593	.15	.38	340	.65	.47	081	.15	.56	811	.65	.67
588	.20	.42	335	.70	.51	076	.20	.60	805	.70	.71
583	.25	.46	330	.75	.55	071	.25	.64	800	.75	.75
578	.30	.51	324	.80	.59	065	.30	.68	794	.80	.79
573	.35	.58	319	.85	.63	060	.35	.72	789	.85	.83
568	.40	.59	314	.90	.68	055	.40	.77	783	.90	.88
563	.45	.63	309	.95	.72	049	.45	.81	778	.95	.92
.97558	20.50	16.67	.97304	23.00	18.76	.97044	25.50	20.85	.96772	28.00	22.96
552	.55	.71	299	.05	.80	039	.55	.89	766	.05	23.00
547	.60	.75	294	.10	.84	033	.60	.93	761	.10	.04
542	.65	.80	289	.15	.88	028	.65	.98	755	.15	.09
537	.70	.84	283	.20	.92	023	.70	21.02	749	.20	.13
532	.75	.88	278	.25	.96	018	.75	.06	744	.25	.17
527	.80	.92	273	.30	19.01	012	.80	.10	738	.30	.21
522	.85	.96	268	.35	.05	007	.85	.14	732	.35	.25
517	.90	17.01	263	.40	.09	001	.90	.19	726	.40	.30
512	.95	.05	258	.45	.13	.96996	.95	.23	721	.45	.34
.97507	21.00	17.09	.97253	23.50	19.17	.96991	26.00	21.27	.96715	28.50	23.38
502	.05	.13	247	.55	.21	986	.05	.31	709	.55	.42
497	.10	.17	242	.60	.25	980	.10	.35	704	.60	.47
492	.15	.22	237	.65	.30	975	.15	.40	698	.65	.51
487	.20	.26	232	.70	.34	969	.20	.44	692	.70	.55
482	.25	.30	227	.75	.38	964	.25	.48	687	.75	.60
477	.30	.34	222	.80	.42	959	.30	.52	681	.80	.64
472	.35	.38	216	.85	.46	953	.35	.56	675	.85	.68
467	.40	.43	211	.90	.51	949	.40	.61	669	.90	.72
562	.45	.47	206	.95	.55	942	.45	.65	664	.95	.77
.97457	21.50	17.51	.97201	24.00	19.59	.96937	26.50	21.69	.96658	29.00	23.81
451	.55	.55	196	.05	.63	932	.55	.73	652	.05	.85
446	.60	.59	191	.10	.67	926	.60	.77	646	.10	.89
441	.65	.63	185	.15	.72	921	.65	.82	640	.15	.94
436	.70	.67	180	.20	.76	915	.70	.86	635	.20	.98
431	.75	.71	175	.25	.80	910	.75	.90	629	.25	24.02
426	.80	.76	170	.30	.84	905	.80	.94	623	.30	.06
421	.85	.80	165	.35	.88	899	.85	.98	617	.35	.10
416	.90	.84	159	.40	.93	894	.90	22.03	611	.40	.15
411	.95	.88	154	.45	.97	888	.95	.07	605	.45	.19
.97406	22.00	17.92	.97149	24.50	20.01	.96883	27.00	22.11	.96600	29.50	24.23
401	.05	.96	144	.55	.05	877	.05	.15	594	.55	.27
396	.10	18.00	139	.60	.09	872	.10	.20	587	.60	.32
391	.15	.05	133	.65	.14	866	.15	.24	582	.65	.36
386	.20	.03	128	.70	.18	861	.20	.28	576	.70	.40
381	.25	.13	123	.75	.22	855	.25	.33	570	.75	.45
375	.30	.17	118	.80	.26	850	.30	.37	564	.80	.49
370	.35	.21	113	.85	.30	844	.35	.41	559	.85	.53
365	.40	.26	107	.90	.35	839	.40	.45	553	.90	.57
360	.45	.30	102	.95	.39	833	.45	.50	547	.95	.62

TABLE III.—*Specific gravity of alcohol by volume at* 60° *F.*—Continued.

Specific gravity at $\frac{60}{60}$° F.	Per cent alcohol by volume.	Per cent alcohol by weight.	Specific gravity at $\frac{60}{60}$° F.	Per cent alcohol by volume.	Per cent alcohol by weight.	Specific gravity at $\frac{60}{60}$° F.	Per cent alcohol by volume.	Per cent alcohol by weight.	Specific gravity at $\frac{60}{60}$° F.	Per cent alcohol by volume.	Per cent alcohol by weight.
.96541	**30.00**	**24.66**	**.96235**	**32.50**	**26.80**	**.95910**	**35.00**	**28.96**	**.95560**	**37.50**	**31.14**
535	.05	.70	229	.55	.84	903	.05	29.00	552	.55	.18
529	.10	.74	222	.60	.89	896	.10	.05	545	.60	.23
523	.15	.79	216	.65	.93	889	.15	.09	538	.65	.27
517	.20	.83	210	.70	.97	883	.20	.13	531	.70	.32
511	.25	.87	204	.75	27.02	876	.25	.18	523	.75	.36
505	.30	.91	197	.80	.06	869	.30	.22	516	.80	.40
499	.35	.95	191	.85	.10	862	.35	.26	509	.85	.45
493	.40	25.00	185	.90	.14	855	.40	.30	502	.90	.49
487	.45	.04	178	.95	.19	848	.45	.35	494	.95	.54
.96481	**30.50**	**25.08**	**.96172**	**33.00**	**27.23**	**.95842**	**35.50**	**29.39**	**.95487**	**38.00**	**31.58**
475	.55	.12	166	.05	.27	835	.55	.43	480	.05	.63
469	.60	.17	159	.10	.32	828	.60	.48	472	.10	.67
463	.65	.21	153	.15	.36	821	.65	.52	465	.15	.72
457	.70	.25	146	.20	.40	814	.70	.57	457	.20	.76
451	.75	.30	140	.25	.45	807	.75	.61	450	.25	.81
445	.80	.34	133	.30	.49	800	.80	.65	442	.30	.85
439	.85	.38	127	.35	.53	794	85	.70	435	.35	.90
433	.90	.42	120	.40	.57	787	.90	.74	427	.40	.94
427	.95	.47	114	.45	.62	780	.95	.79	420	.45	.99
.96421	**31.00**	**25.51**	**.96108**	**33.50**	**27.66**	**.95773**	**36.00**	**29.83**	**.95413**	**8.50**	**32.03**
415	.05	.55	101	.55	.70	766	.05	.87	405	.55	.07
409	.10	.60	095	.60	.75	759	.10	.92	398	.60	.12
403	.15	.64	088	.65	.79	752	.15	.96	390	.65	.16
396	.20	.68	082	.70	.83	745	.20	30.00	383	.70	.20
390	.25	.73	075	.75	.88	738	.25	.05	375	.75	.25
384	.30	.77	069	.80	.92	731	.30	.09	368	.80	.29
378	.35	.81	062	.85	.96	724	.35	.13	360	.85	.33
372	.40	.85	056	.90	28.00	717	.40	.17	353	.90	.37
366	.45	.90	049	.95	.05	710	.45	.22	345	.95	.42
.96360	**31.50**	**25.94**	**.96043**	**34.00**	**28.09**	**.95703**	**36.50**	**30.26**	**.95338**	**39.00**	**32.46**
353	.55	.98	036	.05	.13	695	.55	.30	330	.05	.50
347	.60	26.03	030	.10	.18	688	.60	.35	323	.10	.55
341	.65	.07	023	.15	.22	681	.65	.39	315	.15	.59
335	.70	.11	016	.20	.26	674	.70	.44	307	.20	.64
329	.75	.16	010	.25	.31	667	.75	.48	300	.25	.68
323	.80	.20	003	.30	.35	660	.80	.52	292	.30	.72
316	.85	.24	.95996	.35	.39	653	.85	.57	284	.35	.77
310	.90	.28	990	.40	.43	646	.90	.61	277	.40	.81
304	.95	.33	983	.45	.48	639	.95	.66	269	.45	.86
.96298	**32.00**	**26.37**	**.95977**	**34.50**	**28.52**	**.95632**	**37.00**	**30.70**	**.95262**	**39.50**	**32.90**
292	.05	.41	970	.55	.56	625	.05	.74	254	.55	.95
285	.10	.46	963	.60	.61	618	.10	.79	246	.60	.99
279	.15	.50	957	.65	.65	610	.15	.83	239	.65	33.04
273	.20	.54	950	.70	.70	603	.20	.88	231	.70	.08
267	.25	.59	943	.75	.74	596	.25	.92	223	.75	.13
260	.30	.63	937	.80	.78	589	.30	.96	216	.80	.17
254	.35	.67	930	.85	.83	581	.35	31.01	208	.85	.22
248	.40	.71	923	.90	.87	574	.40	.05	200	.90	.27
241	.45	.76	917	.95	.92	567	.45	.10	193	.95	.31

TABLE III.—*Specific gravity of alcohol by volume at* 60° *F.*—Continued.

Specific gravity at $\frac{60°}{60°}$ F.	Per cent alcohol by volume.	Per cent alcohol by weight.	Specific gravity at $\frac{60°}{60°}$ F.	Per cent alcohol by volume.	Per cent alcohol by weight.	Specific gravity at $\frac{60°}{60°}$ F.	Per cent alcohol by volume.	Per cent alcohol by weight.	Specific gravity at $\frac{60°}{60°}$ F.	Per cent alcohol by volume.	Per cent alcohol by weight.
.95185	**40.00**	**33.35**	**.94786**	**42.50**	**35.58**	**.94364**	**45.00**	**37.84**	**.93916**	**47.50**	**40.13**
177	.05	.39	778	.55	.63	355	.05	.89	906	.55	.18
169	.10	.44	770	.60	.67	346	.10	.93	898	.60	.22
161	.15	.48	761	.65	.72	338	.15	.98	888	.65	.27
154	.20	.53	753	.70	.76	329	.20	38.02	879	.70	.32
146	.25	.57	745	.75	.81	320	.25	.07	870	.75	.37
138	.30	.61	737	.80	.85	311	.30	.12	861	.80	.41
130	.35	.66	729	.85	.90	302	.35	.16	852	.85	.46
122	.40	.70	720	.90	.94	294	.40	.21	842	.90	.51
114	.45	.75	712	.95	.99	285	.45	.25	833	.95	.55
.95107	**40.50**	**33.79**	**.94704**	**43.00**	**36.03**	**.94276**	**45.50**	**38.30**	**.93824**	**48.00**	**40.60**
099	.55	.84	696	.05	.08	267	.55	.35	815	.05	.65
091	.60	.88	687	.10	.12	258	.60	.39	805	.10	.69
083	.65	.93	679	.15	.17	250	.65	.44	796	.15	.74
075	.70	.97	670	.20	.21	241	.70	.48	786	.20	.78
067	.75	34.02	662	.25	.26	232	.75	.53	777	.25	.83
059	.80	.06	654	.30	.30	223	.80	.57	768	.30	.88
052	.85	.11	645	.35	.35	214	.85	.62	758	.35	.92
044	.90	.15	637	.40	.39	206	.90	.66	749	.40	.97
036	.95	.20	628	.45	.44	197	.95	.71	739	.45	41.01
.95028	**41.00**	**34.24**	**.94620**	**43.50**	**36.48**	**.94188**	**46.00**	**38.75**	**.93730**	**48.50**	**41.06**
020	.05	.28	612	.55	.53	179	.05	.80	721	.55	.11
012	.10	.33	603	.60	.57	170	.10	.84	711	.60	.15
004	.15	.37	595	.65	.62	161	.15	.89	702	.65	.20
.94996	.20	.42	586	.70	.66	152	.20	.93	692	.70	.24
988	.25	.46	578	.75	.71	143	.25	.98	683	.75	.29
980	.30	.50	570	.80	.75	134	.30	39.03	679	.80	.34
972	.35	.55	561	.85	.80	125	.35	.07	664	.85	.38
964	.40	.59	553	.90	.84	116	.40	.12	655	.90	.43
956	.45	.64	544	.95	.89	107	.45	.16	645	.95	.47
.94948	**41.50**	**34.68**	**.94536**	**44.00**	**36.93**	**.94098**	**46.50**	**39.21**	**.93636**	**49.00**	**41.52**
940	.55	.73	527	.05	.98	089	.55	.26	626	.05	.57
932	.60	.77	519	.10	37.02	080	.60	.30	617	.10	.61
924	.65	.82	510	.15	.07	071	.65	.35	607	.15	.66
916	.70	.86	502	.20	.11	062	.70	.39	598	.20	.71
908	.75	.91	493	.25	.16	053	.75	.44	588	.25	.76
900	.80	.95	484	.30	.21	044	.80	.49	578	.30	.80
892	.85	35.00	476	.35	.25	035	.85	.53	569	.35	.85
884	.90	.04	467	.40	.30	026	.90	.58	559	.40	.90
876	.95	.09	459	.45	.34	017	.95	.62	550	.45	.94
.94868	**42.00**	**35.13**	**.94450**	**44.50**	**37.39**	**.94008**	**47.00**	**39.67**	**.93540**	**49.50**	**41.99**
860	.05	.18	441	.55	.44	.93999	.05	.72	530	.55	42.04
852	.10	.22	433	.60	.48	990	.10	.76	521	.60	.08
843	.15	.27	424	.65	.53	980	.15	.81	511	.65	.13
835	.20	.31	416	.70	.57	671	.20	.85	502	.70	.18
827	.25	.36	407	.75	.62	962	.25	.90	492	.75	.23
819	.30	.40	398	.80	.66	953	.30	.95	482	.80	.27
811	.35	.45	390	.85	.71	944	.35	.99	473	.85	.32
802	.40	.49	381	.90	.76	934	.40	40.04	463	.90	.37
794	.45	.54	373	.95	.80	925	.45	.08	454	.95	.41

TABLE IV.—*Correction to be applied to the reading of the alcoholometer for different temperatures.*

	° F.	1 P.ct.	2 P.ct.	3 P.ct.	4 P.ct.	5 P.ct.	6 P.ct.	7 P.ct.	8 P.ct.	9 P.ct.	10 P.ct.	11 P.ct.	12 P.ct.	13 P.ct.	14 P.ct.	15 P.ct.
To be added to the observed indication.	50	0.40	0.40	0.40	0.40	0.45	0.50	0.55	0.55	0.60	0.70	0.75	0.85	0.90	1.00	1.10
	51	.35	.35	.35	.35	.40	.45	.50	.50	.55	.65	.70	.75	.80	.90	1.00
	52	.35	.35	.35	.35	.35	.40	.45	.45	.50	.55	.60	.70	.75	.80	.90
	53	.30	.30	.30	.30	.35	.40	.40	.45	.45	.50	.55	.60	.65	.75	.80
	54	.30	.30	.30	.30	.30	.35	.35	.40	.40	.40	.45	.55	.60	.65	.70
	55	.25	.25	.25	.25	.25	.30	.30	.35	.35	.35	.40	.45	.50	.55	.60
	56	.20	.20	.20	.20	.20	.25	.25	.30	.30	.30	.30	.35	.40	.45	.50
	57	.15	.15	.15	.15	.15	.20	.20	.20	.20	.20	.25	.25	.30	.35	.35
	58	.10	.10	.10	.10	.10	.10	.10	.15	.15	.15	.15	.20	.20	.20	.25
	59	.05	.05	.05	.05	.05	.05	.05	.05	.05	.05	.10	.10	.10	.10	.10
To be subtracted from the observed indication.	60	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
	61	.05	.05	.05	.05	.05	.05	.05	.10	.10	.10	.10	.10	.10	.10	.10
	62	.10	.10	.10	.10	.15	.15	.15	.15	.15	.20	.20	.20	.20	.20	.25
	63	.15	.20	.20	.20	.20	.20	.20	.25	.25	.25	.30	.30	.35	.35	.35
	64	.20	.25	.25	.25	.30	.30	.30	.30	.30	.35	.40	.40	.45	.45	.50
	65	.25	.30	.30	.30	.35	.35	.35	.40	.40	.45	.50	.50	.55	.55	.60
	66	.35	.35	.35	.35	.40	.45	.45	.50	.50	.55	.60	.60	.65	.65	.75
	67	.40	.45	.45	.45	.50	.50	.55	.60	.60	.65	.70	.70	.75	.80	.85
	68	.50	.50	.50	.50	.55	.60	.60	.65	.65	.70	.80	.85	.90	.90	1.00
	69	.55	.60	.60	.60	.65	.65	.70	.75	.75	.80	.90	.95	1.00	1.05	1.10
	70	.65	.65	.65	.65	.70	75	.80	.85	.85	.90	1.00	1.05	1.10	1.15	1.25
	71	.75	.75	.75	.75	.80	.85	.90	.95	.95	1.00	1.10	1.20	1.25	1.30	1.40
	72	.80	.80	.80	.85	.90	.95	1.00	1.05	1.05	1.10	1.20	1.30	1.35	1.45	1.55
	73	.90	.90	.90	.90	.95	1.00	1.10	1.15	1.20	1.25	1.35	1.45	1.50	1.55	1.65
	74	.95	.95	.95	1.00	1.05	1.10	1.20	1.25	1.30	1.35	1.45	1.55	1.60	1.70	1.80
	75	1.00	1.05	1.05	1.10	1.15	1.20	1.30	1.35	1.40	1.45	1.55	1.70	1.75	1.85	1.95
	76	.....	1.15	1.15	1.20	1.25	1.30	1.40	1.45	1.50	1.55	1.70	1.85	1.90	2.00	2.10
	77	.....	1.25	1.25	1.30	1.35	1.40	1.50	1.60	1.60	1.65	1.80	1.95	2.05	2.15	2.25
	78	.....	1.30	1.35	1.40	1.45	1.50	1.65	1.70	1.70	1.80	1.95	2.10	2.15	2.25	2.35
	79	.....	1.40	1.45	1.50	1.55	1.60	1.75	1.85	1.85	1.90	2.05	2.20	2.30	2.40	2.50
	80	.....	1.50	1.55	1.60	1.65	1.70	1.85	1.95	1.95	2.00	2.20	2.35	2.45	2.55	2.65

	° F.	16 P.ct.	17 P.ct.	18 P.ct.	19 P.ct.	20 P.ct.	21 P.ct.	22 P.ct.	23 P.ct	24 P.ct.	25 P.ct.	26 P.ct.	27 P.ct.	28 P.ct.	29 P.ct.	30 P.ct.
To be added to the observed indication.	50	1.20	1.30	1.45	1.55	1.70	1.80	1.85	1.95	2.00	2.10	2.20	2.25	2.25	2.30	2.30
	51	1.10	1.20	1.30	1.40	1.65	1.60	1.65	1.75	1.80	1.90	2.00	2.00	2.00	2.05	2.05
	52	1.00	1.05	1.15	1.25	1.35	1.45	1.50	1.55	1.60	1.70	1.75	1.80	1.80	1.85	1.85
	53	.85	.95	1.05	1.10	1.20	1.25	1.30	1.40	1.40	1.45	1.55	1.55	1.60	1.60	1.60
	54	.75	.80	.90	.95	1.00	1.10	1.15	1.20	1.20	1.25	1.30	1.35	1.35	1.40	1.40
	55	.65	.70	.75	.80	.85	.90	.95	1.00	1.00	1.05	1.10	1.10	1.10	1.15	1.15
	56	.50	.55	.60	.65	.70	.70	.75	.80	.80	.85	.90	.90	.90	.90	.90
	57	.40	.40	.45	.50	.50	.55	.55	.60	.60	.65	.65	.65	.65	.70	.70
	58	.25	.30	.30	.30	.35	.35	.40	.40	.40	.40	.45	.45	.45	.45	.45
	59	.15	.15	.15	.15	.15	.20	.20	.20	.20	.20	.20	.20	.20	.25	.25
To be subtracted from the observed indication.	60	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
	61	.15	.15	.15	.15	.15	.20	.20	.20	.20	.20	.20	.20	.20	.20	.25
	62	.25	.30	.30	.30	.35	.35	.40	.40	.40	.40	.40	.40	.45	.45	.45
	63	.40	.40	.40	.45	.50	.55	.55	.55	.65	.65	.65	.65	.65	.65	.70
	64	.50	.55	.55	.60	.70	.70	.75	.75	.85	.85	.85	.85	.90	.90	.90
	65	.65	.70	.70	.75	.85	.90	.95	.95	1.05	1.05	1.05	1.05	1.10	1.10	1.15
	66	.80	.85	.85	.90	1.00	1.05	1.15	1.15	1.25	1.25	1.25	1.25	1.30	1.30	1.35
	67	.95	1.00	1.00	1.05	1.15	1.25	1.30	1.35	1.45	1.45	1.45	1.45	1.50	1.55	1.60
	68	1.05	1.10	1.20	1.25	1.35	1.40	1.50	1.55	1.65	1.70	1.70	1.70	1.75	1.75	1.80
	69	1.20	1.25	1.35	1.40	1.50	1.60	1.65	1.75	1.85	1.90	1.90	1.90	1.95	2.00	2.05
	70	1.35	1.40	1.55	1.55	1.65	1.75	1.85	1.95	2.05	2.10	2.10	2.10	2.15	2.20	2.25
	71	1.50	1.55	1.65	1.70	1.80	1.90	2.05	2.15	2.25	2.30	2.30	2.35	2.35	2.40	2.45
	72	1.65	1.75	1.80	1.85	1.95	2.10	2.20	2.35	2.45	2.50	2.55	2.55	2.60	2.65	2.70
	73	1.75	1.85	1.85	2.05	2.15	2.25	2.40	2.55	2.60	2.75	2.75	2.80	2.80	2.85	2.90
	74	1.85	2.00	2.10	2.20	2.30	2.45	2.55	2.75	2.80	2.95	3.00	3.00	3.05	3.10	3.15
	75	2.05	2.15	2.25	2.35	2.45	2.60	2.75	2.95	3.00	3.15	3.20	3.25	3.25	3.30	3.35
	76	2.20	2.30	2.40	2.55	2.65	2.75	2.95	3.10	3.20	3.35	3.40	3.45	3.50	3.50	3.55
	77	2.35	2.45	2.60	2.70	2.80	2.95	3.10	3.30	3.40	3.55	3.65	3.70	3.70	3.75	3.80
	78	2.50	2.65	2.75	2.90	3.00	3.10	3.30	3.45	3.60	3.75	3.85	3.90	3.95	3.95	4.00
	79	2.65	2.80	2.95	3.05	3.15	3.30	3.45	3.65	3.80	3.95	4.10	4.15	4.15	4.20	4.25
	80	2.80	2.95	3.10	3.25	3.35	3.45	3.65	3.80	4.00	4.15	4.30	4.35	4.40	4.40	4.45

DETERMINATION OF THE ALCOHOL.

1. Fill the measuring cylinder to the upper mark with the liquid to be tested (having previously ascertained the temperature of the liquid and the reading of the Balling saccharometer in the liquid), the bottom of the meniscus[1] being on a level with the mark, then empty the measured liquid into the still, letting it drain as completely as possible, then fill the cylinder with one-half of the original volume (the lower mark) with water, and rinse it out, pouring the water into the contents of the still. The measuring of both volumes must be exact.

2. Close the still and make all the connections tight, fill the condenser with cold water, and light the alcohol lamp.

3. Continue the distillation until the original volume taken has been collected in the receiver. Put out the light. Mix the contents of the receiver thoroughly. Insert the alcoholometer and take the reading of the thermometer and hydrometer. Be careful not to carry the distillation further than filling the receiver to the mark. The distillation may be stopped one or two drops before the distillate has reached the mark and the volume brought to the mark with water.

4. If the distillation is hurried so that the liquid foams and the distillate becomes colored the operation must be stopped, the still and condenser thoroughly cleaned, and another volume of the liquid taken. It will generally be found that after half the liquid is distilled the distillate will become cloudy or milky, but such cloudiness does not affect the specific gravity essentially.

5. Take the reading of the alcoholometer and thermometer, and correct the reading of the former for the observed temperature according to Table IV. Then:

Corrected reading of the alcoholometer multiplied by the specific gravity of absolute alcohol corresponding to this reading, as found in Table III, and the product divided by the specific gravity of the wine, corresponding to the corrected reading of the Balling saccharometer as found in Table II, equals the percentage by volume of the alcohol in the original wine.

METHODS OF SUGAR ANALYSIS.

By the term "sugar analysis" it is understood that the analyses of all the common saccharine substances, viz, juices, sugar, molasses, sirup, starch sugar, and honey are embraced. In the following directions an attempt will be made to indicate the general principles which should guide the practice of sugar analysis, together with sufficient detail to enable these analyses to be carried out with accuracy and uniformity.

The principal components to be determined in the substances mentioned above are:

1. Water.
2. Density and total solids.
3. Ash.
4. Nitrogenous bodies.
5. Reducing sugar.
6. Sucrose.
7. Lactose.
8. Starch sugar.
9. Raffinose.

WATER.

In sugar or melada.—Weigh from 2 to 5 grs. into a flat dish (nickel or platinum) and dry at the temperature of boiling water for three hours; cool in a desiccator and weigh; return to the oven and dry for an hour. If on weighing there is only a slight

[1] The surface of the liquid as seen from above is not quite level, as it rises at the side of the flask and around the hydrometer stem. The rising at the sides give the surface a crescent shape, hence the name "meniscus." The horns of the crescent rise above the level surface of the liquid, hence the direction to look across the bottom of the meniscus, where the horns do not prevent a clear view of the level portion of the surface.

change of weight, the process may be considered finished; otherwise the drying must be continued until the loss of water in one hour is not greater than 0.20 per cent.

In honey or molasses.—Proceed as above, using only 1 to 2 grs. of substance.

DENSITY AND TOTAL SOLIDS.

The density of juices, sirups, etc., is most conveniently determined by means of Beaumé's or Brix's hydrometer or areometer, preferably with the latter, as the graduations of the scale give close approximations to the percentages of total solids. The Brix spindle should be graduated to tenths, the divisions of the scale being of sufficient length to enable the analyst to note exactly the point to which the stem sinks in the liquid. It is therefore desirable, for accuracy, that the range of degrees recorded by each individual spindle be as limited as possible, this end being best secured by the employment of sets, consisting of not less than three spindles. The solutions should be as nearly as possible of the same temperature as the air at the time of reading, and if the variation from the standard temperature of the graduation of the spindle amounts to more than 1° C., compensation therefor must be made by reference to the table of corrections for temperature on page 114, Tucker's Sugar Analysis.

Care should be observed, in taking the density of a juice, to allow it to stand in the cylinder until all air bubbles have escaped, before reading the spindle. When it is desired to accurately determine the specific gravity of the sample, a specific-gravity bottle or picnometer of 50 cc. capacity is employed, and the weight of the amount required to fill the bottle at 15.5° C., divided by 50, gives its correct specific gravity. When the temperature varies from the standard temperature for specific gravities, the weight of the juice must be compared with an equal volume of water weighed at the same temperature.

The per cent of *total solids* can be approximately ascertained directly from the reading of the Brix hydrometer; or, knowing the reading of the Beaumé spindle, or the specific gravity of the sample, by reference to the table on pages 116–118 Tucker's Sugar Analysis, the desired percentage can be obtained.

In order to accurately estimate the proportion of total solids in a juice or sirup, from 1 to 3 grs. (according to the density of the liquid) are run into a porcelain dish or glass schaelchen, containing about 2 grs. of finely powdered ignited silica. The whole is then placed on a water bath and heated until the contents are apparently dry, after which it is transferred to an air bath and dried to a constant weight at a temperature not exceeding 110° C. The weight of the dish and silica subtracted from the weight of the dish and contents after drying, gives total solids.

ASH.

(*a*) Take 5 or 10 grs. of the material (sugar, molasses, honey) in a platinum dish[1] of 50 to 100 cc. capacity; heat at 100° C. until the water is expelled, and then slowly until intumescence ceases. The dish is then placed in a muffle and heated at low redness until a white ash is obtained.

(*b*) *Soluble ash.*—Digest the ash with water, filter through a Gooch crucible, wash with hot water, dry the residue at 100° C., and weigh. The difference of weights equals soluble ash.

(*c*) Use 50 mgs. of zinc oxide to 25 grs. of molasses or 50 grs. of sugar. Incoporate thoroughly by adding dilute alcohol, and mixing; dry, and ignite as above. Deduct the weight of zinc used from the weight of ash.

(*d*) Carbonize the mass at a low heat, dissolve the soluble salts with hot water, burn the residual mass as above, add the solution of soluble salts, and evaporate to dryness at 100° C.; ignite gently, cool in a desiccator, and weigh.

(*e*) Saturate the sample with H_2SO_4, dry, ignite gently, then burn in a muffle at a low redness.

[1] If the substance contains tin or any other metal capable of uniting with platinum, a dish made of some other material must be used.

(*f*) Dissolve 10 grs. of sugar in hot water and filter to remove sand, etc.; evaporate the filtrate and washings to dryness; carefully carbonize, and extract with hot water until there is no longer any reaction for chlorine. Dry and ignite the residue; weigh as insoluble ash. Add the soluble portion, treat with HCl in slight excess, and evaporate to dryness. Heat until all free HCl is driven off; take up with H_2O and a little HCl, filter and wash. The residue is silica. Add NH_4OH to the filtrate, boil and filter, wash the residue, which is iron and alumina. To the filtrate add $(NH_4)_2C_2O_4$; evaporate to dryness; ignite and moisten with $(NH_4)_2CO_3$ and re-ignite; dissolve in water, filter, and wash. The residue on the filter represents carbonates of lime and magnesia. Evaporate the filtrate to a small bulk, add $(NH_4)_2CO_3$, and evaporate to dryness; drive off the excess of ammonia cautiously and weigh. This gives the alkalis as carbonates, and this weight, added to the insoluble ash, formerly determined, gives the total carbonate ash.

ALTERNATE METHOD.

To avoid the correction of one-tenth, as proposed by Scheibler, and one-fifth as proposed by Girard and Violette, when sugars are burned with H_2SO_4, Boyer suggests incineration with benzoic acid, as giving the real quantity of mineral matter without correction.

The benzoic acid is dissolved in alcohol of 90 per cent, 25 grs. of the acid to 100 cc. of alcohol; 5 grs. of the sugar is weighed in a capsule and moistened with 1 cc. of H_2O. The capsule is heated slowly, in order to caramelize the sugar without carbonizing it; 2 cc. of the benzoic acid is next added, and the capsule warmed until all the alcohol is evaporated; the temperature is then elevated until the sugar is converted into carbon. The decomposing benzoic acid produces abundant vapors which render the mass extremely porous, especially if a circular motion is imparted to the capsule. The slow heating is continued until all the benzoic acid is volatilized.

The carbon obtained is voluminous and of a brilliant black color. The incineration is accomplished in a muffle at a low red heat. The capsule should be weighed quickly when taken from the desiccator, in order to avoid the absorption of water by the alkaline carbonates. Benzoate of ammonia may be employed instead of benzoic acid, and the analyst should previously assure himself that neither the acid nor the ammonia salt leaves a residue on incineration. In addition to giving the mineral matters directly, this method permits the determination of their composition also, a matter of no small importance.

NITROGENOUS BODIES.

Any of the methods adopted by the association for the estimation of nitrogen may be used.

REDUCING SUGAR.

Reagent, alkaline copper solution. (*Fehling, Violette.*)

34.64 grs. of pure crystallized copper sulphate.
187.00 grs. of tartrate of soda and potash.
78.00 grs. of caustic of soda.

Dissolve the $CuSO_4+5H_2O$ in water, and make up to 1,000 cc. at the average temperature at which the analyses are to be made. Mix the solutions of Rochelle salts and caustic soda, and make up to 1,000 cc. In practice use equal volumes of each reagent.

Processes (*volumetric*).—(*a*) The solution to be examined should contain about 1 per cent of reducing sugar. To 10 cc. of the copper solution and 10 cc. of the alkali, in a large test tube or casserole, add 20 cc. of water and boil. Add the sugar solution in approximately the proper quantity to reduce the copper, and boil for exactly two minutes. If, on standing for a moment, the solution is still blue, add more sugar solution and re-boil. When no longer able to distinguish any residual copper by the eye, suck a few drops of the boiling hot liquid into a Wiley-Knorr end reaction tube, and test the filtrate for Cu with ferrocyanide of potassium solution and acetic acid.

If preferred the Ross method, using filter paper, may be used.[1] If copper be still present, add the sugar solution little by little, boil, and re-test until no copper is found. Having thus determined the approximate quantity of sugar solution to be used, repeat the test twice, adding immediately each time almost the entire quantity of sugar solution necessary to complete reduction.

(*b*) *Gravimetric.*—Take 25 cc. of copper solution of the following composition:

34.639 grs. of $CuSO_4+5H_2O$ in 500 cc. of water, add in an Erlenmeyer flask 25 cc. of alkali solution made as follows:

173 grs. of Rochelle salts
51.7 grs. of NaOH
} in 500 cc. of water.

Add 50 cc. of a sugar solution of such strength as to leave a part of the copper unreduced; heat quickly to the boiling point, and boil for exactly two minutes; add 100 cc. of recently boiled, cold, distilled water. The liquid is poured at once into a filter tube made of hard glass, with asbestos diaphragm (an ordinary extraction tube is well adapted for this purpose). The asbestos film should be previously moistened. Bring finally all the separated suboxide into the tube with the help of a feather brush, and wash with 300 to 400 cc. of boiling water, afterward with 20 cc. of absolute alcohol, and finally with ether; then dry, heat to low redness to convert into oxide, and to destroy any organic matter. The copper may also be weighed in the metallic state after reduction in a stream of dry hydrogen. The tube should in all cases be kept in a desiccator before weighing.

(*c*) (*Gravimetric.*)

Reagents:

34.639 grs. of $CuSO_4+5H_2O$
125.000 grs. of NaOH
173.000 grs. of Rochelle salts
} in 500 cc. of water.

Process.—In a beaker of 250 cc. capacity place 25 cc. each of the reagents, and 50 cc. of water. Heat to boiling and add 25 cc. of the solution to be tested, which should contain about 1 per cent of reducing sugar (dextrose, levulose). Boil for exactly two minutes, decant into a Gooch crucible, wash by decantation with 100 cc. of boiling-hot water, keeping the cuprous oxide in the beaker constantly covered. Place the beaker containing the cuprous oxide in place of the one receiving the wash water and dissolve the Cu_2O on the Gooch crucible in a fine stream of HNO_3; wash until all $Cu(NO_3)_2$ is removed. Transfer the whole of the nitrate from the beaker to a tared platinum dish, add a little H_2SO_4, and evaporate over a steam bath until all HNO_3 is expelled, and the Cu is converted into $CuSO_4$; connect with a battery of four gravity cells, and allow the electrolytic action to continue for three hours, or until all the Cu is deposited. (Connect the dish with the zinc pole of the battery.) Pour off the acid solution, adding water until every trace of H_2SO_4 is removed; wash with 95 per cent alcohol, and finally with absolute alcohol; ignite the residual alcohol, cool in a desiccator, and weigh; calculate the sugar reduced from the weight of metallic copper by the following factors:

(1) Multiply the weight of copper by 0.5698 for the weight of invert sugar.

(2) Multiply the weight of copper by 0.5808 for the weight of anhydrous dextrose.

Sucrose.—(Optical method, to be used when the sugar is approximately pure.

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[1] A small filter, the diameter of which need not exceed 2 inches, is folded as for ordinary filtrations, and is immersed, point down, in the hot supernatant liquid, the apex of the filter being just beneath the surface of the solution.

A portion of the liquid at once filters through, and is removed from the interior of the filter by means of a small dropping pipette, consisting of a small glass tube with finely drawn out point and provided with a rubber bulb at the upper extremity.

The liquid is readily taken up and expelled from the pipette by alternately compressing and relaxing pressure upon the bulb.

A fresh filter is used for each filtration, and the pipette is rinsed after each portion of the solution is tested.

METHOD OF THE U. S. INTERNAL REVENUE OFFICE.[1]

A very high degree of accuracy may be obtained in the optical estimation of sugars, if the proper conditions are observed. Such conditions are (1) accurately graded and adjusted instruments, weights, flasks, tubes, etc.; (2) skilled and practiced observers; (3) a proper arrangement of the laboratories in which the work is performed, and (4) a close adherence to the most approved methods of manipulation.

On the other hand, if due observance is not paid to these conditions, the sources of error are numerous, and inaccurate results inevitable.

We will endeavor to point out in this report the best means of meeting the proper conditions for obtaining the highest degree of accuracy consistent with fairly rapid work. It would be manifestly impossible to observe too great a refinement of accuracy in this work, such as would be employed in exact scientific research. This would be unnecessary for the end in view, and impossible on account of the amount of time that would be required.

I. Instruments and Apparatus.

It is of the greatest importance that the polariscopes and all apparatus used in the work shall be carefully and accurately adjusted and graduated, and upon a single and uniform system of standardization. Recent investigations of the polarimetric work done in the customs branch of the Treasury Department have shown, that a very considerable part of the want of agreement in the results obtained at the different ports was due to a lack of uniformity in the standardization of the instruments and apparatus.

(*a*) *The polariscope.*—There are many different forms of this instrument used. Some are adapted for use with ordinary white light, and some with monochromatic light, such as that of the sodium ray. They are graduated and adjusted upon various standards, all more or less arbitrary. Some, for example, have their scales based upon the displacement of the polarized ray produced by a quartz plate of a certain thickness; others upon the displacement produced by an arbitrary quantity of pure sucrose, dissolved and made up to a certain volume and polarized in a certain definite length of column. It would be very desirable to have an absolute standard established for polariscopic measurements, to which all instruments could be referred, and in the terms of which all such work could be stated. This commission has information that an investigation is now in progress, under the direction of the German Imperial Government, having for its end and purpose the determination of such data as will serve for the establishment of such an absolute standard. When this is accomplished, it can easily be made a matter of international agreement, and all future forms of instruments could then be based upon it. This commission would suggest that the attention of the proper authorities be called to the desirability of official action by this Government looking to its cooperation with other countries for the adoption of international standards for polarimetric work. Until this is done, however, it will be necessary for the Internal Revenue Bureau to adopt, provisionally, one of the best existing forms of polariscopes, and, by carefully defining the scale of this instrument, establish a basis for its polarimetric work which will be a close approximation to an absolute standard, and upon which it can rely in case of any dispute arising as to the results obtained by the officers of the Bureau.

For the instrument to be provisionally adopted by the Internal Revenue Bureau, this commission would recommend the half-shadow instrument made by Franz Schmidt and Haensch, of Berlin. This instrument is adapted for use with white-light illumination from coal-oil or gas lamps. It is convenient and easy to read, requiring no delicate discrimination of colors by the observer, and can be used even by a per-

[1] From the report of a scientific commission appointed by the U. S. Bureau of Internal Revenue for the purpose of establishing standards and methods for the polarimetric estimation of sugars, to be used in ascertaining the bounty to be paid upon sugars of domestic production and for the purpose of securing uniformity in such standards and methods.

son who is color blind. This form of instrument is adjusted to the Ventzke scale, which, for the purposes of this report, is defined to be such that 1 degree of the scale is the one-hundredth part of the rotation produced in the plane of polarization of white light in a column 200 mm. long, by a standard solution of chemically pure sucrose at 17°. 5 C., the standard solution of sucrose in distilled water being such as to contain, at 17°. 5 C. in 100 cc., 26.048 grs. of sucrose.

In this definition the weights and volumes are to be considered as absolute, all weighings being referred to a vacuum.

The definition should properly be supplemented with a statement of the equivalent circular rotation in degrees, minutes, and seconds, that would be produced by the standard solution of sugar used to read 100° on the scale. This constant is now a matter of investigation, and it is thought best not to give any of the hitherto accepted values. When this is established, it is recommended that it be incorporated in a revision of the regulations of the Internal Revenue relative to sugar, in order to make still more definite and exact the official definition of the Ventzke scale.

The instruments should be adjusted by means of control quartz plates, three different plates being used for complete adjustment, one reading approximately 100° on the scale, one 90° and one 80°.

These control quartz plates should have their exact values ascertained, in terms of the Ventzke scale, by the Office of Weights and Measures, by comparison with the standard quartz plates in possession of that office, in strict accordance with the foregoing definition, and should also be accompanied by tables giving their values for temperatures from 10° C. to 35° C.

(*b*) *Weights.*—The weights used should be of solid brass, and should be standardized by the Office of Weights and Measures.

(*c*) *Flask.*—The flasks used should be of such a capacity as to contain at 17°.5 C. 100.06 cc., when filled in such a manner that the lowest point of the meniscus of the surface of the liquid just touches the graduation mark. The flasks will be standardized to contain this volume, in order that the results shall conform to the scale recommended for adoption, without numerical reduction of the weighings to vacuo. They should be calibrated by the Office of Weights and Measures.

(*d*) *Tubes.*—The tubes used should be of brass or glass, 200 and 100 millimeters in length, and should be measured by the Office of Weights and Measures.

(*e*) *Balances.*—The balances used should be sensitive to at least one milligram

II. Skilled Observers.

The commission recommends that the work of polarizing sugars be placed in the hands of chemists, or at least of persons who are familiar with the use of the polariscope, and have some knowledge of the theory of its construction and of chemical manipulations. To this end we would suggest that applicants for positions where such work is to be done should be obliged to undergo a competitive examination in order to test their fitness for the work that is to be required of them.

III. Arrangement of Laboratories.

The arrangement of the rooms in which polarizations are performed has an important bearing upon the accuracy of the results obtained.

Polariscopic observations are made more readily and accurately if the eye of the observer is screened from diffused light; therefore, a partial darkening of the room, which may be accomplished by means of curtains or hangings, is an advantage. On the other hand, the temperature at which the observation is made, has a very considerable influence upon the results obtained, so that the arrangements for darkening the room must not be such as will interfere with its proper ventilation. Otherwise the heat from the lamps used, if confined within a small room, will cause considerable variations in the temperature of the room from time to time.

The proper conditions will best be met, in our opinion, by placing the lamps either in a separate room from that in which the instruments are, and perforating the wall

or partition between the two rooms for the light to reach the end of the instruments, or in a ventilated hood with the walls perforated in a like manner. By lining the wall or partition on both sides with abestos paper, and inserting a plate of plane glass in the aperture through which the light passes, the increase of temperature from the radiation of the lamp will be still further avoided. With the lamps separated from the instruments in this manner, the space in which the instruments are contained is readily darkened without much danger of its temperature being unduly raised.

Some light, of course, is necessary for reading the scales, and if artificial light is employed for this purpose, the sources chosen should be such that as little heat as possible will be generated by them. Small incandescent electric lights are best for this purpose. Refinements of this kind can not always be used, of course, but the prime requisite with reference to the avoidance of temperature errors is that all operations—filling the flasks and tubes, reading the solutions, controlling the instrument with standard quartz plates, etc.—should be done at one and the same temperature, and that this temperature be a constant one, that is, not varying greatly at different hours of the day. For example, the room should not be allowed to become cold at night, so that it is at low temperature in the morning when work is begun, and then rapidly heated up during the day. The polariscope should not be exposed to the direct rays of the sun during part of the day, and should not be near artificial sources of heat, such as steam boilers, furnaces, flues, etc.

The tables upon which the instruments stand should be level.

IV. Methods of Manipulation.

The methods of manipulation used in the polarization of sugar are of prime importance. They consist in weighing out the sugar, dissolving it, clarifying the solution, making it up to standard volume, filtering, filling the observation tube, regulating the illumination, and making the polariscopic reading.

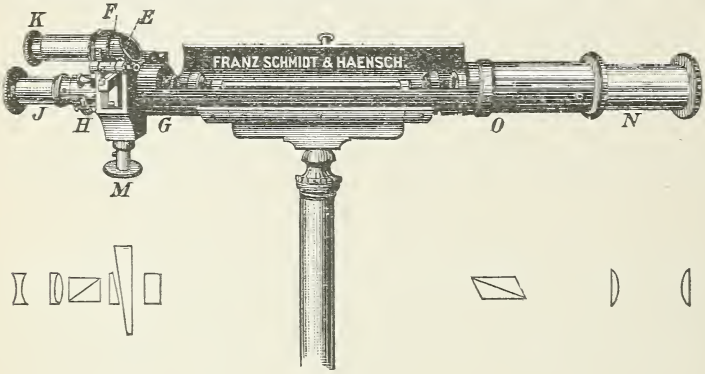

Fig. 7.

The proper conduct of these processes, in connection with the use of accurately graduated apparatus, is the only surety against the numerous sources of error which may be encountered. Different sugars require different treatment in clarification, and much must necessarily be left to the judgment and experience of the operator.

The following directions are based upon various official procedures, such as the one used in the United States custom-houses, the method prescribed by the German government, etc. They embody also the results of recent research in regard to sources of error in the polarimetric estimation of sugar:

Directions for the Polarization of Sugar.

I. *Description of Instrument and Manner of Using.*

The instrument employed is known as the half-shadow apparatus of Schmidt and Haensch. It is shown in Fig. 7.

The tube N contains the illuminating system of lenses and is placed next to the lamp; the polarizing prism is at O and the analyzing prism at H. The quartz wedge compensating system is contained in the portions of the tube marked F, E, G, and is controlled by the milled head M. The tube J carries a small telescope, through which the field of the instrument is viewed, and just above is the reading tube K, which is provided with a mirror and magnifying lens for reading the scale.

The tube containing the sugar solution is shown in position in the trough between the two ends of the instrument. In using the instrument, the lamp is placed at a distance of at least 200 millimeters from the end; the observer seats himself at the opposite end in such a manner as to bring his eye in line with the tube J. The telescope is moved in or out until the proper focus is secured, so as to give a clearly defined image, when the field of the instrument will appear as a round, luminous disk, divided into two halves by a vertical line passing through the center, and darker on one half of the disk than on the other. If the observer, still looking through the telescope, will now grasp the milled head M and rotate it, first one way and then the other, he will find that the appearance of the field changes, and at a certain point the dark half becomes light and the light half dark. By rotating the milled head delicately backwards and forwards over this point, he will be able to find the exact position of the quartz wedge operated by it, in which the field is neutral or of the same intensity of light on both halves.

The three different appearances presented by the field are best shown in the following diagram:

FIG. 8.

With the milled head set at the point which gives the appearance of the middle disk as shown above, the eye of the observer is raised to the reading tube K, and the position of the scale is noted. It will be seen that the scale proper is attached to the quartz wedge, which is moved by the milled head, and attached to the other quartz wedge is a small scale called a vernier, which is fixed, serving for the exact determination of the movable scale with reference to it. On each side of the zero line of the vernier a space corresponding to nine divisions of the movable scale is divided into ten equal parts. By this device the fractional part of a degree indicated by the position of the zero line is ascertained in tenths; it is only necessary to count from zero, until a line is found which makes a continuous line with one on the movable scale.

With the neutral field as indicated above, the zero of the movable scale should correspond closely with the zero of the vernier, unless the zero point is out of adjustment.

If the observer desires to secure an exact adjustment of the zero of the scale, or in any case if the latter deviates more than one-half of a degree, the zero lines are made to coincide by moving the milled head and securing a neutral field at this point by means of the small key which comes with the instrument, and which fits a small nipple on the left hand side of F, the fixed quartz wedge of the compensating system. This nipple must not be confounded with a similar nipple on the right hand side of the analyzing prism H, which it fits as well, but which must never be touched, as the adjustment of the instrument would be seriously disturbed by moving it. With the key on the proper nipple it is turned one way or the other until the field is neutral. Unless the deviation of the zero be greater than 0.5°, it will not be necessary to use the key, but only to note the amount of the deviation, and for this purpose the observer must not be content with a single setting, but must perform the

operation five or six times, and take the mean of these different readings. If one or more of the readings show a deviation of more than 0.3° from the general average, they should be rejected as incorrect. Between each observation the eye should be allowed ten to twenty seconds of rest.

The "setting" of the zero having been performed as above, the determination of the accurate adjustment of the instrument by means of the "control" quartz plates is proceeded with. Three such plates will be furnished with each polariscope, which have "sugar values," respectively, approximating 80°, 90°, and 100°. These values vary with the temperature, and tables are furnished with them giving their exact value at different temperatures from 10° to 35° C.

One of these plates is placed in the instrument and the field observed; it will be seen that the uniform appearance of the field is changed. The milled head is turned to the right until the exact point of neutrality is re-established, just described above in setting the zero. The scale is read, the observation repeated, the reading taken again, and so on until five or six readings have been made. The average is taken, readings being rejected which show a divergence of more than 0.3°, and the result corrected for the deviation of the zero point, if any was found, the deviation being added if it was to the left and subtracted if to the right. If the adjustment of the instrument be correct, the result should be the value of the control plate used, as ascertained from the table for the temperature of 20° C. Each of the three plates is read in the instrument in this way. A variation of 0.3° from the established values may be allowed for errors of observation, temperature, etc., but in the hands of a careful observer a deviation greater than this with one of the three plates after a careful setting of the zero, shows that the instrument is not accurately adjusted.

The complete verification of the accurate adjustment of the polariscope by means of three control plates, as given above, should be employed whenever it is set up for the first time by the officers using it, whenever it has sustained any serious shock or injury, and whenever it has been transported from one place to another. It should also be done at least once a week while the instrument is in active use.

After the complete verification has been performed as described, further checking of the instrument is done by means of one control plate alone, the one approximating 90°, and the setting of the zero point is dispensed with, the indication of the scale for sugar solutions being corrected by the amount of deviation shown in the reading of the 90° control plate from its established value, as ascertained from the table at the temperature of the room.

For example, a sugar solution polarizes 80.5°; the control plate just before had given a polarization of 91.4°, the temperature of the room during both observations being 25° C. According to the table the value of the control plate at 25° C. is 91.7°; the reading is therefore 0.3° too low and 0.3° is added to the reading of the sugar solution, making the corrected result 80.8°. The temperature of the room should be ascertained from a standardized thermometer placed close to the instrument and in such a position as to be subject to the same conditions. In closing this report the members of this Commission hereby signify their intention to promote uniformity and accuracy by adopting and using the standards and general plan of procedure recommended in this report in the polarimetric determinations over which, in their respective branches of Government work, they have control.

Respectfully submitted.

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Optical method by inversion.—For raw sugars, molasses, etc.

(1) *Method of Clerget.*—Make up the solution as above, and place 50 cc. of the filtrate in a flask marked at 50 and 55 cc. Fill to the upper mark with HCl of 1,188 sp. gr.,

and mix well; place in hot water and heat quickly to 68° C.; keep at this temperature for exactly ten minutes; remove, cool quickly to room temperature, and polarize, noting the temperature. If the sample contained originally any invert sugar the second polarization should be made at approximately the same temperature as the first. The percentage of sucrose is then calculated by the following formula:

S = percentage of sucrose.
a = first polarization.
b = second polarization (usually to the left).
$a + b$ = sum of the polarizations.
t = temperature of observation in degrees C.

$$\text{then } S = \frac{a + b}{^{1}142.4 - \frac{t}{2}}$$

When b is to the left, a and b are to be added; when b is to the right, it is to be subtracted from a.

(2) *Method of Lindet.*—Place 50 cc. of the filtrate as above in a flask marked at 100.5 cc.; add 5 gr. of powdered zinc (zinc dust), place in boiling water, and when at the boiling point add, little by little, 5 cc. of strongest HCl. After the acid has all been added, cool and make up to mark. Polarize in a 400-mm. tube, or multiply the reading in a 200-mm. tube by 2. Calculate the percentage of sucrose as before.

Gravimetric method.—Determine first any reducing sugar in the sample by the methods mentioned above; then invert sucrose, neutralize free acid, and redetermine the reducing sugar. Deduct the percentage of reducing sugar obtained at first, and the remainder will be reducing sugar derived from sucrose; multiply this number by 95 to obtain the percentage of sucrose in the sample.

ELECTIVE METHODS.

The elective methods remain the same as in Bulletin 28, pp. 217–228.

METHODS FOR SOIL ANALYSIS.

(1) *Gathering samples.*—The soil selected should be as far as possible in its natural condition, not modified by recent applications of manure, or changed by the transporting action of water or wind. Surface accumulations of decaying leaves, etc., should be removed before taking the sample.

To eliminate accidental variations in the soil, select specimens from five or six places in the field which seem to be fair averages of the soil, remove 2 or 3 pounds of the soil, taking it down to the depth of 6 to 9 inches so as to include the whole depth of the soil. Mix these soils intimately, remove any stones, shake out all roots and foreign matter, and dry the soil until it becomes friable. Break down any lumps in a mortar with a wooden pestle, but avoid pulverizing any mineral fragments; pass 8 to 10 pounds of the soil through a sieve, having circular perforations one twenty-fifth of an inch in diameter, rejecting all pebbles and materials too coarse to pass through the sieve. Once more mix intimately the sifted soil. Expose in thin layers in a warm room till thoroughly air-dry (or dry it in an air-bath at a temperature of 40° C.), place 6 to 8 pounds in a clean bottle, with label of locality and date, and cork the bottle containing the soil, for analysis.

The soil is rapidly dried to arrest nitrification; it is not heated above 40° lest there be dissipation of ammonia compounds, or a change in the solubility of the soil. The normal limit to which the soil may be heated in place by the sun's rays should not be exceeded in preparing a soil for an agricultural chemical analysis.

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[1] This factor, 142.4, is the one adopted provisionally by the association instead of 144 given by Clerget.

The relative amount of fragments too coarse to pass through the sieve should be made a matter of record. They are soil material, but not yet soil, so far as agricultural purposes are concerned.

(2) *Relation of the soil to water.*—This belongs to physics rather than chemistry; yet the mutual relation and interaction of soil and water are so intimately associated with the chemical activity of the soil as to demand consideration by the agricultural chemist.

(3) *Hygroscopic moisture.*—Place 5 grs. of the air-dry soil in a flat-bottomed and tared plantinum dish; heat in an air-bath to 110° C. for eight hours; cool in a desiccator, and weigh; repeat the heating, cooling and weighing at intervals of an hour, till a constant weight is obtained, and estimate the hygroscopic moisture by the loss of weight. Weigh rapidly to avoid absorption of moisture from the air. The loss of weight × 20 = per cent of hygroscopic moisture in the air-dry soil.

(4) *Capacity to hold water.*—The power of a soil to retain water is an important element of fertility. It can be approximately determined with rapidity by the following process. In the throat of a clean 3-inch glass funnel place a very small filter, just large enough to prevent the soil from running through the stem; wet the filter, and pour into the glass funnel 100 grs. of the air-dry soil; from a graduated foot glass, containing 100 cc. of water, pour water on the soil till it is thoroughly wet and a few drops of water pass through the soil; let it stand undisturbed till no more water flows from the soil, the funnel being covered with a glass plate to prevent evaporation. Return the water which has filtered through the soil to the graduate. The number of cubic centimeters taken up by the soil will show the percentage capacity of the soil to hold water.

In using this process certain precautions are necessary to secure uniform results. The soil should be simply poured into the funnel, and not pressed or compacted in any way. When water is poured upon the soil, no disturbing influence, no handling or shaking of the soil, should take place. Let the adhesion between the water and soil, and the pull of gravitation settle the whole question, while the results are measured by the cubic centimeters of the water retained in the soil.

It is only claimed that the results are approximate, but the approximation is so close, and the process so rapid, especially where many soils are to be compared, that it is hoped the method may receive a trial.

(5) *Capillary power of soils.*—This is usually determined by filling a long glass tube with soil, placing the lower end in water, and marking the height to which the water ascends, as shown by the changed color of the soil in the tube. The results are, confessedly, unsatisfactory. It takes weeks, and sometimes months, for the water to reach its final elevation, or the column of soil becomes separated by an air-space, and the experiment fails.

Is not such an experiment an inversion of nature's method? For agricultural purposes water comes, primarily, from above, and then sinks through the soil. If long glass tubes are filled with dry soil, and water applied at the top till the soil in the tube is filled with water, the natural process is more closely followed. By noting how fast the water descends we get some notion of the drainage capacity of the soil. If the lower end of the soil tube is securely capped by a cloth bound over the end so as to prevent the soil from escaping, and is then placed in a deep reservoir of water so that we may raise or lower the tube without lifting it out of the water, by then shifting the position of the vertical tube we may at last find a certain elevation of the top of the tube above the water level in the reservoir, where the soil will just remain moist, but not wet, and thus measure the agricultural capillarity of the soil.

(6) *Volatile matter.*—The platinum crucible and 5 grs. of soil (4) used to determine the hygroscopic moisture may be used to determine the volatile matter. Heat the crucible and dry soil to low redness in a muffle furnace, or the sheet-iron-cone furnace, to be described for incinerations of organic substances in ash analysis. The heating should be prolonged till all organic material is burned away, but below the temperature at

which alkaline chlorides would volatilize. The residue, when cooled, is usually of a reddish color, from the presence of ferric oxide. Moisten the cold mass with a few drops of a saturated solution of carbonate of ammonia, dry it, and heat to 150° C. in an air bath to expel excess of ammonia. The loss in weight of the dry soil (4) represents organic matter, water of combination, salts of ammonia, etc. These are all entered on record as volatile matter.

(7) *Water-soluble materials of the soil.*—To prepare a water extract of the soil, the cylindrical glass percolator of the druggist may be employed. Satisfactory results have been secured in this laboratory by using a tin percolator made as follows: A straight cylinder of the best sheet tin, 30 c. long and 7 c. in diameter, is closed at one end by a flat cap of tin. In the center of the cap a cone of tin is soldered, 1.5 c. in diameter where it enters the cap, 5 mm. at the other end, and 20 c. long. All parts are securely soldered, to make the percolator water-tight and strong. The tin cone is made long and slim, so as to easily penetrate a perforated rubber cork in case the filter pump is required.

To use this tin percolator for making a soil extract, place a disk of two thicknesses of filter paper of the diameter of the cylinder upon the inside of the cap, over this a disk of perforated tin, and on top of this a disk of thick filter paper, and pour into the percolator a kilogram of the sifted air-dry soil. If the soil is sandy and porous, press it down, to make it more retentive of water; otherwise the sifted soil should be simply shaken to settle it.

By means of a perforated rubber cork connect the neck of the percolator with a two-necked bottle of two liters capacity, pour sufficient distilled water (ammonia free) on the soil to moisten it all, and let the whole stand undisturbed for half an hour, then add more pure distilled water, and if the filtration is too slow use the filter pump, till a liter of filtrate is secured. If the soil extract is cloudy, filter through a plain filter. Each cubic centimeter of filtrate contains the water-soluble materials of 1 gr. of air-dry soil.

(8) *Soluble solids.*—Evaporate 100 cc. to dryness on the water bath in a tared dish to determine the percentage of water-soluble materials in the soil; each gram of residue representing 1 per cent of such materials. Test this dry residue for nitrates by pouring over it 10 cc. of pure H_2SO_4, holding in solution 3 or 4 milligrams of sulphate of brucia.

(9) *Chlorides.*—Titrate 100 cc. with standard decinormal argentic nitrate with two drops of a solution of K_2CrO_4 as an indicator. Titrate in a white porcelain dish and view the reaction through a yellow-glass plate of such tint as will eliminate the color of the chromic solution. The reaction will then be sharply defined. More than one part of soluble chlorides in one thousand of soil is injurious to agricultural plants.

(10) *Sulphates.*—Precipitate the soluble sulphates in 100 cc. with $BaCl_2$ in presence a few drops of HCl, and estimate the soluble sulphates.

Reserve the rest of the water solution (8) for the estimation of nitrates (26).

ACID-SOLUBLE MATERIALS.

In the following scheme for soil analysis it is recommended to use the air-dry soil from the sample bottle for each separate investigation. A determination made once for all of hygroscopic moisture and of water of combination on a separate specimen of air-dry soil, will afford corrections for all the other samples used. It is not desirable to ignite the soil before analysis, or to heat it so as to change its chemical properties.

In an agricultural chemical analysis the object is to find the kind and quantity of soil materials available for the growing plant. The reserve or inactive materials are not objects of immediate concern. Prof. Hilgard takes pure HCl of the specific gravity 1.115 as the solvent for soil materials, which may be supposed to fully represent the solvent action that may be secured by water and other solvents in the soil, and the action of the roots of growing plants, or other corroding agents. The same acid and

of the same strength is here recommended, but with a modified form of using the same. Instead of digesting the soil with this acid in a covered beaker, with liability to continual variation of strength of the acid, it is proposed to use the acid with constant strength, except so far as it may be neutralized by combination with the soil minerals.

Instead of a porcelain beaker covered with a watch glass, I propose for the soil digestion a 4-ounce vial of Bohemian glass, with a flat-topped ground-glass stopper. The small steam bath is a copper vessel 8 inches in diameter and 5 inches deep, with vertical sides; the cover has four openings, through which the vials may readily pass down $2\frac{3}{4}$ inches to a perforated false bottom upon which the vials rest, and the space of $2\frac{1}{4}$ inches below the false bottom serves for the hot-water chamber of the steam bath. Through the center of the cover and of the false bottom a vertical tube, open at both ends, extends nearly to the bottom of the vessel, for pouring in water to replenish the waste; or a side tube near the bottom of the hot-water chamber may be connected with a water reservoir to keep the water in the steam bath at a constant level. A Bunsen burner serves to heat the steam bath and to keep the body of the vials at the constant heat of boiling water. When such a bath is set to work the digestion can go forward day and night with very little care and attention. If the vials charged for digestion are placed in the apparatus when cold, and then heated up to steam heat, no trouble is found from the vials breaking. When the vials are properly charged and the escape of acid prevented, thirty-six to forty hours of continuous digestion will be as effective as five days of intermittent digestion in a covered beaker.

(11) *Acid digestion of the soil.*—Weigh 5 grs. of the air-dry soil into a 4-ounce Bohemian vial, add 50 cc. of pure HCl sp. gr. 1.115, insert the glass stopper, wire it securely, place in the steam bath, and digest for thirty-six to forty hours at the temperature of boiling water. Pour the contents of the vial into a small beaker, wash out the vial with distilled water, add the washings to the contents of the beaker, pour into this 2 cc. of HNO_3 to peroxidize the iron and oxidize organic matter, and evaporate the contents of the beaker to complete dryness over the water bath. Cool the beaker, add 10 cc. of pure HCl sp. gr. 1.115 and 50 cc. of distilled water, and heat to near boiling. Filter from sand and silica, wash the filter with distilled water till the filtrate shows no reaction with silver nitrate, and make the filtrate up to 500 cc. (Solution A), 100 cc. of which represents the soluble materials from 1 gr. of air-dry soil.

(12) *Sand and silica.*—Dry the filter and insoluble residue from A, transfer the residue to a tared platinum dish, burn the filter and add its ash to the dish, heat the dish and contents, at first gently to avoid spurting of silica, then intensely to destroy organic matter, cool in a desiccator and weigh. The increase of weight, minus the filter ash, represents the sand and silica. Boil this residue for fifteen minutes in 50 cc. of a strong solution of sodic carbonate, add 100 cc. of boiling water, filter while still hot, and wash the filter and contents with boiling water till the sodic salts are washed away. Dry the residue, burn the filter and add its ash to the insoluble residue, heat this to redness, cool, and weigh. Deduct the ash of the filter and enter the balance as sand or insoluble silicates. The difference in weight between sand, and sand and silica, enter as silica. The sand and silica will respectively represent the amount of these materials in 5 grs. of air-dry soil, and these weights multiplied by 20 will give the per cent respectively of sand and silica in such soil.

(13) *Ferric oxide and alumina.*—To 200 cc. of Solution A (in an Erlenmeyer flask), add NH_4HO to alkaline reaction (avoiding excess), to precipitate ferric and aluminic oxides and phosphates. Expel the excess of ammonia by boiling, allow to settle, decant the clear solution through a filter; add to the flask 50 cc. of hot distilled water, boil, settle, and decant as before. After pouring off all the clear solution possible, dissolve the residue with a few drops of HCl with heat, and add just enough NH_4HO to precipitate the oxides. Wash by decantation with 50 cc. of distilled water, and then transfer all the precipitate to the filter, and wash with hot distilled water till the

filtrate becomes free from chlorides (Save the filtrate and washings, Solution B.) Dry the filter and precipitate in the air bath at 110° C., and transfer the precipitate to a tared platinum crucible, burn the filter, and add the ash to the precipitate, heat the whole red hot, cool in a desiccator and weigh. The increase of weight, minus the ash of the filter, and the phosphoric acid (found in a separate process), represents the weight of the ferric and aluminic oxides.

(14) *Ferric oxide.*—Place the whole of the ignited oxides in an Erlenmeyer flask (200 cc. capacity), add 10 cc. of conc. H_2SO_4, and digest on the steam bath till complete solution is effected[1]; cool and add 100 cc. of distilled water, a piece of amalgamated zinc and a slip of platinum foil, cover with a watch glass, and allow to stand for twenty-four hours to reduce ferric to ferrous salt. When the reduction is complete, as tested by transferring on a glass rod a drop of the solution with a drop of ammonic sulpho-cyanide on a white porcelain surface, pour the solution at once into a beaker, wash out the flask and transfer the washings to the beaker, taking special pains to exclude any zinc, mercury, or other reducing agents, add 2 cc. of H_2SO_4, make up the solution to 250 cc. with pure recently boiled water, and titrate with standard solution of permanganate for the ferric oxide present in 2 grs. of air-dry soil.

Preparation of standard permanganate solution.—Dissolve 3.156 grs. of pure crystallized permanganate of potassium in 1,000 cc. of distilled water at 16° C., and preserve this in a ground-glass-stoppered bottle, shielded from the light. Standardize this solution with pure ferrous sulphate, or ammonic-ferrous sulphate, or oxalic acid according to directions in Johnson; Fresenius, section 112; or Sutton, Volumetric Analysis, section 30; and determine the equivalent weight of Fe_2O_3 for each cubic centimeter of the permanganate solution.

The weight of ferric oxide deducted from ferric oxide and alumina (14), with corrections for filter ash and phosphoric acid, will give the weight of alumina in 2 grs. of air-dry soil.

(15) *Manganese.*—Concentrate the filtrate of washings from B to 200 cc. If a qualitative test of the soil shows the presence of maganese, add a few drops of bromine to the solution till the color becomes orange, and keep the solution at the temperature of 60° C. for twenty-four hours. The maganese will separate as a brownish hydrate, $Mn_2C(OH)_2$. Filter, wash the precipitate, dry, and heat to redness, weigh and estimate as Mn_3O_4.

(16) *Lime.*—If no manganese is precipitated, add to solution B, or otherwise to the filtrate and washings (from 15), 20 cc. of a strong solution of NH_4Cl and 40 cc. of a saturated solution of $(NH_4)_2C_2O_4$ to completely precipitate all the lime as oxalate, and convert the magnesia into soluble magnesic oxalate. Heat to boiling and let stand for six hours till the calcic oxalate settles clear, decant the clear solution onto a filter, pour 50 cc. of hot distilled water on the precipitate, and again decant the clear solution onto the filter, transfer the precipitate to the filter, and wash it free from all traces of oxalates and chlorides. Place the funnel over the mouth of a 500 cc. Erlenmeyer flask, puncture the apex of the filter with a glass rod, wash the oxalate into the flask with a jet of water, dissolve any adhering oxalate from the filter by dilute H_2So_4 (10 per cent solution), wash the filter with a stream of distilled water, add to the flask 20 cc. of H_2SO_4, make the volume up to 300 cc., heat to 70° C., and titrate with a standard solution of permanganate of such strength that one cubic centimeter will be decolorized by 0.0063 gr. of crystallized oxalic acid. Each cubic centimeter of permanganate solution will represent 0.0028 gr. of CaO.

(17) *Alternate method.*—Transfer the washed and dried oxalate to a tared platinum crucible, burn the filter on the crucible cover, add the ash to the precipitate, cover this with conc. H_2SO_4, heat gently to dryness, and then intensely, to expel the excess of

[1] If the ignited oxides do not dissolve readily in H_2SO_4, dissolve them in a few drops of HCl. Expel the excess of acid, add 10 cc. of H_2SO_4, and proceed as directed in the text.

H_2SO_4, cool in a desiccator, and weigh. Estimate the increase of weight, minus filter ash, as calcium sulphate, $CaSO_4 \times 0.41158 = CaO$.

(18) *Second alternate method.*—Transfer the precipitate to a tared platinum crucible, burn the filter and add this to the precipitate, heat the crucible and contents to a low red heat to burn the oxalate. Moisten the cooled mass with a saturated solution of ammonic carbonate, dry, and heat cautiously to a low red heat, cool and weigh. The increase of weight (minus filter ash) represents calcic carbonate, $CaCO_3 \times 0.56 = CaO$.

(19) *Magnesia.*—Concentrate the filtrate and washings (from 17) to 200 cc., place in a half-liter Erlenmeyer flask, add 30 cc. of a saturated solution of Na_2HPO_4 and 20 cc. of conc. NH_4HO, cork the flask, and shake violently at intervals of a few minutes till crystals form, then set the flask in a cool place for twelve hours. Filter off the clear liquid through a tared Gooch filter, transfer the precipitate to the filter, and wash with dilute ammonic hydrate (1:3) till the filtrate is free from phosphates; dry and ignite the crucible, at first gently and then intensely, to form magnesium pyrophosphate. The increase of weight $\times 0.36024 = MgO$. By using an Erlenmeyer flask free from scratches and marks, and shaking violently instead of stirring with a glass rod, the danger is almost entirely avoided of crystals adhering to the sides of the vessel; but if crystals do adhere they are as readily removed by a rubber-tipped glass rod from an Erlenmeyer flask as from a beaker.

(20) *Sulphuric acid.*—Evaporate 200 cc. of solution A (12) nearly to dryness on a water bath to expel the excess of acid; then add 100 cc. of distilled water; heat to boiling and add 10 cc. of a solution of $BaCl_2$, and continue the boiling for five minutes. When the precipitate has settled pour the clear liquid on a tared Gooch filter, treat the precipitate with 50 cc. of boiling water, and transfer the precipitate to the filter and wash with boiling water till the filtrate is free from chlorides. Dry the filter and ignite strongly. The increase in weight is barium sulphate, which $\times 0.34331 = SO_3$ in 2 grs. of air-dry soil.

(21) *Phosphoric acid.*—To the filtrate and washings from (20) add NH_4HO to alkaline reaction, then $(NH_4)_2CO_3$ and a few drops of $(NH_4)_2C_2O_4$ to complete precipitation; boil, settle, and decant the clear solution on a filter, add boiling water to the precipitate and again decant; finally bring the precipitate on the filter and wash thoroughly. Dissolve the precipitate in HNO_3, and add molybdate of ammonium in excess to the solution. Keep at a temperature of 70° C. for six hours, and from the phospho-molybdate of ammonium, estimate the phosphoric acid in the usual way.

The material used in the estimation of ferric oxide and alumina (14) may also serve for a separate estimation of P_2O_5. After titration with permanganate, heat the solution to boiling and precipitate with NH_4HO. Wash the precipitate by decantation, dissolve in hot HNO_3 and precipitate by ammonic molybdate as before, and estimate as pyrophosphate of magnesia. The pyrophosphate $\times 0.6396 = P_2O_5$. In estimating the alumina in the mixed precipitate of ferric and aluminic oxides and phosphates, the P_2O_5 must be subtracted to obtain the final weight of alumina. Thus, from the final weight of the precipitate by NH_4HO (14), subtract the filter ash, the ferric oxide as determined by titration, then the P_2O_5, and the remainder will be Al_2O_3.

The solubility of the phosphates in the soil is intimately related to their availability for growing crops. It has been assumed that phosphates soluble in acetic acid are active and immediately available for crops, and that soils containing acetic-soluble phosphates will not be benefited by the use of superphosphates. To determine the solubility of soil phosphates, boil 10 grs. of soil in 50 cc. of strong acetic acid for fifteen minutes, filter, evaporate the filtrate to dryness, ignite, dissolve the residue in HNO_3 with heat, and test the solution with excess of molybdate of ammonium at 70° C.

(22) *Potash and soda.*—Evaporate the filtrate and washings (from 21) to dryness, heat to a low red heat to decompose oxalates and expel ammonia salts, dissolve in 25 cc. of distilled water, filter and wash the precipitate, add to the filtrate and washings

10 cc. of baryta water, and digest for an hour. Filter and wash the precipitate, add ammonic carbonate to the filtrate to complete precipitation of baryta, filter and wash this precipate. Evaporate the filtrate and washings in a tared platinum dish, gently ignite the residue to expel ammonic salts, cool and weigh. The increase of weight represents the chlorides of potassium and sodium in 2 grs. of air-dry soil.

Separate and estimate the potassium chloride by platinic chloride according to the official method of the Official Association of Agricultural Chemists.

Subtract the weight of potassium chloride as thus found, from the weight of potassium chloride and sodium chloride. The difference represents sodium chloride.

Alternate method.—For an alternate method for alkalis, use J. Lawrence Smith's method as given in Crook's Select Methods, second edition, pp. 28 to 40.

(23) *Other alkali metals.*—The salts of lithium, cæsium, and rubidium are occasionally found in very small amounts in soils. The agricultural uses of these salts are still in question, and their amount is too small to admit of quantitative estimation. A qualitative examination may be made by the spectroscope with the water-soluble materials (8) evaporated to dryness and dissolved with 2 or 3 drops of HCl. Test by the spectroscope with a platinum wire in a Bunsen flame.

(24) *Nitrogen of the soil.*—The combined nitrogen in the soil, and the state of combination in which it is held, are subjects of great importance to the agricultural chemist. The nitrogen compounds in the soil are usually placed in three classes:

1. The nitrogen combined with oxygen as nitrates or nitrites, existing as soluble salts in the soil.

2. The nitrogen combined with hydrogen as ammonia, or organic nitrogen easily convertible into ammonia. The ammonia may exist as salts, or be occluded by hydrated ferric or aluminic oxides and organic matter in the soil.

3. The inert nitrogen of the soil or the humus nitrogen.

The nitrogen in the first and second classes is considered the active nitrogen of the soil, so far as plant food is concerned, while the inert nitrogen is, for the time being, incapable of affording sustenance to agricultural plants, and hence is properly placed in a class by itself. But the exchanges between the first and second classes are well known to chemists; the reduction of nitrates to ammonia, and the oxidation of ammonia to nitrates are familiar to agricultural chemists. It has also been a matter of discussion which of these forms is better fitted to nourish plant life. They seem to have equal agricultural activity, and their exchanges are matters of almost daily occurrence. Why should they be separately estimated in an agricultural chemical analysis? Why not class them together as active soil nitrogen, and estimate their amount in one operation?

25. *Active soil nitrogen.*—The material proposed for reducing the nitrates to ammonia, and at the same time to bring ammonia salts and organic nitrogen into a condition for separation by distillation, is sodium amalgam. Liquid sodium amalgam may be readily prepared by placing 100 cc. of mercury in a flask of half a liter capacity, covering the warmed mercury with melted paraffin and dropping into the flask at short intervals pieces of metallic sodium the size of a large pea (taking care that the violence of the reaction does not project the contents from the flask), till 6.75 grs. of sodium have combined with the mercury. The amalgam contains 0.5 per cent of sodium and may be preserved indefinitely under the covering of paraffin. The mercury is easily recovered at the close of the operation, and nothing of value is wasted except the sodium.

To estimate the active soil nitrogen, weigh 50 grs. of air-dry soil and place it in a clean mortar. Take 200 cc. of ammonia-free distilled water, rub up the soil with a part of the water to a smooth paste, transfer this to a flask of 1 liter capacity, washing the last traces of the soil into the flask with the rest of the water. Add 25 cc. of the liquid sodium amalgam and shake the flask so as to break the sodium amalgam into small globules distributed through the soil. Insert a stopper with a Kroonig valve, and set aside in a cool place for twenty-four hours. Pour into the flask 50 cc. of

milk of lime, and distill on a sand bath 100 cc. into a flask containing 20 cc. of decinormal sulphuric acid, and titrate with decinormal soda solution, using dimethyl-anilin orange as an indicator. Estimate the nitrogen of the ammonia found as active soil nitrogen. If the ammonia produced is too small in amount to be readily estimated volumetrically, determine the ammonia by nesslerizing the distillate.

26. *Estimation of nitrates in the soil.*—When it is desired to estimate separately the nitrates in the soil the following modification of (25) may be used: Evaporate 100 cc. of the soil extract (8) to dryness on the water bath; dissolve the soluble portion of the residue in 100 cc. of ammonia-free distilled water, filtering out any insoluble residue, place the solution in a flask and add 10 cc. of liquid sodium amalgam, insert the stopper with Kroonig valve, set it aside to digest in a cool place for twenty-four hours, add 50 cc. of milk of lime, distill and titrate as in (25), and estimate the nitrogen as N_2O_5.

Nesslerizing may be substituted for titration when the amount of nitrates is small.

An approximate estimation of the amount of nitrates will be of value in determining which method of estimation to use. This may be done by evaporating a measured quantity of the soil extract (8), say 5 cc. more or less, on a porcelain cover on a steam bath or radiator, having first dissolved a minute fragment of pure sulphate of brucia in the soil extract. When dry, pour over the residue concentrated sulphuric acid free from nitrates, and observe the color reactions produced.

If the nitrate (reckoned as KNO_3) left upon evaporating the quantity of water taken does not exceed two one-thousandths of a milligram, only a pink color will be developed by adding the sulphuric acid; with three one-thousandths of a milligram, a pink with faint reddish lines; with four one-thousandths, a reddish color; with five one-thousandths, a red color.

By increasing or diminishing the amount of soil extract evaporated to secure a color reaction of a certain intensity, an approximate estimate may be made of the amount of nitrates present.

Blank experiments to test the acid and the brucine will be required before confidence can be placed in such an estimation.

27. *Total nitrogen of soils.*—The total nitrogen of soils may be determined by the usual combustion with soda-lime, but this process is often unsatisfactory because of the large amount of material required when the organic matter or humus is small in amount.

A modification of the Kjeldahl method is more easy to carry out and gives results equally satisfactory. Weigh out 20 grs. of air-dry soil, place this in a Kjeldahl flask, and pour in 20 cc. of sulphuric acid (free from ammonia), holding in solution 1 gr. of salicylic acid. (If the soil contains much lime or magnesia in the form of carbonate, enough more sulphuric acid must be added to secure a strongly acid condition of the contents of the flask.) Add gradually 2 grs. of zinc dust, shaking the contents of the flask to secure intimate mixture. Place the flask in a sand bath and heat till the acid boils, and maintain the boiling for ten minutes. Add 1 gr. of mercury and continue the boiling for one hour, adding 10 cc. of sulphuric acid if the contents of the flask are likely to become solid. Cool the flask and wash out the soluble materials in the flask with 200 cc. of pure water, leaving the heavy earthy materials in the Kjeldahl flask. Rinse the residue with 100 cc. of water and add this to the first washings. Place this soluble acid extract in a liter digestion flask, add 35 cc. of a solution of potassium sulphide, and shake the flask to secure intimate mixture of the contents. Introduce a few fragments of granulated zinc, pour in 75 cc. of a saturated solution of caustic soda, connect the flask with a condenser and distill 150 cc. into a flask containing 20 cc. of decinormal sulphuric acid, and titrate with decinormal soda solution, using cochineal or dimethyl-anilin orange as an indicator.

Enter the nitrogen found in this operation as total soil nitrogen.

The difference between the total soil nitrogen and the active soil nitrogen will express the inert nitrogen of the soil.

(28) *Acid soils.*—Soils of good agricultural quality are usually neutral or slightly alkaline, but soils are found which give a decidedly acid reaction when blue litmus paper is pressed upon the moist surface. Swamp muck is often acid from the presence of humic acid. Drying the muck removes the acid quality by rendering the muck insoluble in water. If an acid soil becomes neutral by drying, and the water filtered through the dried soil is free from acidity, it is probable that the acid condition was caused by an organic acid of the humus class. But if the acid condition persists after drying the soil, the cause is to be sought in sulphates of some heavy metal, *e. g.*, iron or copper, the sulphates of which have an acid reaction.

METHODS FOR ASH ANALYSIS.

PREPARATION OF ASH.

Before combustion the material must be thoroughly cleaned from all foreign matter; especially from adhering soil, wood, bark, roots, etc., by brushing and dusting, wiping with a moist sponge, and finally by rubbing gently with a soft cotton cloth; seeds, by placing on a fine sieve and drenching them with distilled water with constant shaking till the water runs off clear, and finally rubbing the seeds between a soft cotton cloth. The material should then be dried to a constant weight at the temperature of boiling water.

COMBUSTION OF ORGANIC SUBSTANCES.

The combustion should be carried on at a comparatively low temperature, never reaching a full red heat, because of the danger of volatilizing alkaline chlorides, etc., nor in a strong draft of air, lest the lighter parts of the ash, *e. g.*, silica, be carried away.

Combustion is best carried on in a flat platinum dish in a cast-iron muffle, 18 inches long, 3½ inches high, and 5 inches wide at the bottom, the muffle resting on a firebrick inside the furnace to moderate the bottom heat, and the fuel piled upon the top and sides of the muffle to burn the material by the surface heat.

When this Lawes & Gilbert muffle is not at command, the ordinary assay furnace may be used by placing a firebrick under the muffle, placing the platinum dish and material for combustion near the middle of the muffle, feeding the furnace with fuel on the top and sides of the muffle so as to maintain it at a low red heat, and leaving out the plug of the muffle so as to allow a very slow draft of air.

When no muffle of any kind is available, the substance may be burned to ash in a platinum dish properly guarded. In place of a muffle use the sheet-iron dish commonly employed for a 4-inch sand bath. Place the empty sheet-iron dish on an iron tripod or other support, so that the gas flame from a Bunsen burner may cover the whole bottom of the dish. On this dish place a sheet-iron cone (of Russia iron), 6 inches high, 3½ inches in diameter at the bottom, and 1 inch at the top. Such a dish-and-cone cover approximates the condition of a muffle for materials placed inside the cone.

For the incineration use a flat-bottomed platinum dish 3 inches in diameter and 1 inch deep. Place the material for combustion in the platinum dish, put this in the empty sheet-iron dish, place over the platinum dish and inside the sheet-iron dish the sheet-iron cone, and heat the sheet-iron dish to a low red heat by a gas flame. The cone should be made of Russia sheet iron to avoid the danger of scales of iron rust falling into the ash during combustion.

The cost of such a combustion apparatus is small and the manipulation simple, the platinum dish and contents will not be heated to volatilization of alkaline chlorides, there will not be sufficient draft of air to carry away any ash, yet the heat within the cone will slowly and surely incinerate the contents of the dish.

With substances rich in silica and alkalis it is better to first char the substance. Wash with distilled water to remove soluble salts, then dry and incinerate the residue. Evaporate the watery extract and add this to the rest of the ash.

With substances rich in phosphates, *e. g.*, seeds and animal substances, char the material and remove salts by acetic acid, decant the acetic solution, wash with distilled water, and then complete the combustion. Add the acetic solution and washings to the final ash, evaporate to dryness, and gently ignite the whole to decompose the acetates. By this method seeds, etc., may be incinerated in eight to ten hours.

In whatever way obtained, the whole of the ash should be pulverized and intimately mixed before analysis.

ANALYSIS OF WOOD ASHES.

Weigh out 100 grs. of the air-dry ash, and pass through a wire sieve (20 meshes to the inch) to separate materials manifestly foreign, *e. g.*, nails, broken glass and pottery, pebbles, etc., and estimate the per cent of such accidental materials. Pulverize any charcoal and semifused portions of ash remaining on the sieve, sift them and mix intimately with the sifted ash, and preserve in stoppered bottles for analysis.

1. *Moisture.*—Weigh out 5 grs. of the ash in a tared platinum dish and heat to 110° C., in an air bath to constant weight. Cool in a desiccator and weigh. The loss of weight $\times$ 20 = per cent of moisture in the ash.

2. *Carbon.*—Heat this dried ash in a platinum dish in the sheet-iron-and-cone apparatus described for incinerating organic substances, till the ash is uniformly grayish-white and there is no further loss of weight; weigh and determine this loss of weight, which $\times$ 20 = per cent of charcoal in original ash.

3. *Sand and silica.*—Place this ignited ash in a 4-ounce glass-stoppered vial; measure out 50 cc. of HCl (sp. gr. 1.115) and pour onto the ash cautiously to prevent loss of ash by spurting, and when all effervescence has ceased, add the balance of the acid: insert the glass stopper, wire it securely, and place in a steam bath (described under soil analysis) for two hours; empty the vial into a platinum dish, wash the vial with distilled water, adding the washings to the ash solution, and evaporate the whole to dryness on a water bath. Add 10 cc. of dilute HCl and 50 cc. of distilled water to the contents of the platinum dish, transfer the contents to a Schleicher & Schuell filter, wash with distilled water till the last drops of the filtrate are free from chlorides (when tested by solution of $AgNO_3$), dry and ignite the precipitate and filter. If there are no grains of sand (revealed by grittiness when stirred with a glass rod), subtract the ash of the filter from the weight of this residue and estimate the balance as silica. If sand is present, boil the ignited and weighed residue in a strong solution of Na_2CO_3 to dissolve silica, wash by decantation to remove all soda salts, dry and weigh the sand, the difference between the weight of sand, and silica + sand, will give the weight of silica, and this $\times$ 20 = per cent of silica in the ash.

4. *Phosphoric acid.*—Evaporate the acid filtrate and washings from the silica to 100 cc., place 40 cc. of this in an Erlenmeyer flask of 250 cc. capacity, add NH_4HO till nearly neutralized, then add 30 cc. of citro-magnesic[1] mixture, then 30 cc. of concentrated NH_4HO, cork the flask and shake violently at intervals of a few minutes till crystallization is well established. Set the flask in a cool place for four hours, then filter the $MgNH_4PO_4$ on a tared Gooch filter, wash the precipitate with dilute ammonia (1 to 3), dry the Gooch filter, ignite, at first gently and then intensely, to form pyrophosphate of magnesia. The increase of weight of the Gooch filter equals the pyrophosphate of magnesia from 5 grs. of ash. This multiplied by 5.1168 ($6396 \times 80 = 5.1168$) will give the percentage of P_2O_5 in the ash.

In this method it is important to remove all the silica before precipitating the phosphoric acid. It is also essential to use so much of the citro-magnesic mixture as to

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[1] The citro-magnesic mixture is prepared by dissolving 270 grs. of citric acid in 350 cc. of warm water and adding, by degrees, 27 grs. of $MgCO_3$. When effervescence ceases and the liquid is cool, add 400 cc. of dilute (1 to 10) ammonic hydrate, and dilute the whole to a liter. Preserve in a well-stoppered bottle. In the presence of a large excess of NH_3, 20 cc. of this mixture will insure the precipitation of one decigram of P_2O_5 even in the presence of ferric and aluminic salts, unless their quantity is excessive.

See Sutton's Volumetric Analysis, 5th edition, page 239.

prevent the precipitation of phosphate of iron or alumina. If the addition of the citro-magnesic mixture causes an immediate precipitation, the precipitate is ferric or aluminic phosphate, and not enough of the citro-magnesic mixture was used. In this case the process must be renewed from the separation of silica, and the amount of citro-magnesic mixture increased till no precipitate forms immediately after its addition. In this case the addition of NH_4HO in excess will cause the complete precipitation of phosphate of magnesia and ammonia after a time, while ferric and aluminic salts will be held in solution.

Alternate method.—Molybdate of ammonia. The official method for analysis of insoluble phosphates, as prescribed for determination of total phosphoric acid, is recommended as the alternate method.

5. *Carbonic acid.*—Heat 4 or 5 grs. of ash in the sheet-iron-and-cone muffle till all charcoal is consumed; cool in a desiccator, weigh out 2 grs. of ash and transfer to a Schroetter alkalimeter. Fill one chamber of the alkalimeter with HNO_3 (sp. gr. 1.2) and the other with conc. H_2SO_4 to dry the escaping CO_2. Wipe the outside of the alkalimeter from every trace of dust and moisture, and weigh the apparatus. Open the stopcock of the HNO_3 chamber and permit the acid to flow so as to decompose the ash slowly, the CO_2 bubbling up, a bubble at a time, through the H_2SO_4. When effervescence ceases let the whole of HNO_3 flow into the reservoir below, attach a $CaCl_2$ tube to the top of the HNO_3 chamber, heat the alkalimeter on a sand bath to gentle ebullition, and suck dry air through the apparatus till the CO_2 is removed. Set aside the alkalimeter till it becomes cold, and then weigh the apparatus. The loss of weight will represent the weight of CO_2 in 2 grs. of ash.

Alternate method.—By Liebig's potash bulbs. The usual process of absorption by solution of KHO, weighing, etc.

6. *Chlorine.*—Pour out the nitric solution from the alkalimeter upon a filter, wash out the last traces of the solution, pass the soluble matters through the filter, and wash the insoluble residue with water acidulated with HNO_3. To this filtrate add solution of $AgNO_3$ to complete precipitation of the chlorides, boil and stir with a glass rod till the silver chloride becomes flocculent, let it settle, decant the clear liquid upon a filter, add 100 cc. of water acidulated with HNO_3 and heat to boiling; again decant the clear liquid upon the filter and wash the precipitate with boiling distilled water; finally bring the precipitate upon the filter and wash with distilled water till the filtrate gives no reaction with dilute HCl. Dry the precipitate thoroughly and transfer to a tared porcelain crucible with cover; ignite the filter on the crucible cover, moisten the ash with a drop of HCl, evaporate the excess of acid, place the lid on the crucible, and heat the crucible till the silver chloride begins to melt around the edges. Cool the crucible and weigh. The increase of weight (minus the filter ash) multiplied by 0.1236 (*i. e.* $\frac{35.46}{143.43} \times 50$) will give the per cent of chlorine in the ash.

This process should be carried on in the absence of direct sunlight.

Alternate method.—Boil 10 grs. of ash in 400 cc. of pure water for half an hour; transfer all to a measuring flask of 500 cc. capacity, wash the beaker and add the washings to the flask, cool, make up the volume to 500 cc. and mix intimately; filter off through a dry filter 100 cc., add a drop of solution of phenol-phthalein, and neutralize with dilute HNO_3 till only a faint pink color remains, add 2 drops of a strong solution of K_2CrO_4 and titrate with standard decinormal solution of $AgNO_3$ (16.956 grs. $AgNO_3$ to 1,000 cc.) Every cubic centimeter of the standard silver solution equals 0.003546 gr. of chlorine in 2 grs. of ash, or cc. $\times$ 0.1773 = per cent of chlorine in ash.

In performing this titration, watch the reaction through a plate of amber-colored glass of such tint as will neutralize the color of potassic chromate; the reaction to form silver chromate then becomes sharply defined.

The reliability of this method will depend upon the accuracy with which neutralization by nitric acid has been made. The least trace of free acid or alkaline carbonate will vitiate the results.

Second alternate method.—To 100 cc. of the solution in the foregoing method add HNO_3 to strong acid reaction, then solution of $AgNO_3$ to complete precipitation, and then proceed as in the first method for estimating chlorine.

These alternate methods are based on the assumption that boiling water will dissolve all the chlorides present in wood ashes.

7. *Sulphuric acid* —Place 5 grs. of ash in a digestion vial. Measure out 50 cc. of HCl (sp. gr. 1.115) and cautiously pour the acid on the ash till effervescence ceases, then pour in the rest of the acid, place the glass stopper in place and wire it securely, and place the vial in the digestion steam bath for two hours. Pour the contents of the vial into a 250 cc. measuring flash, wash out the vial and add the washings to the flask, cool, make up to 250 cc. with distilled water and mix intimately. Filter through a dry filter 100 cc. into a beaker and evaporate on the water bath till the excess of acid is expelled; add 100 cc. of distilled water, heat to boiling temperature and precipitate with $BaCl_2$ in excess. Let it stand for twelve hours in a warm place, then decant the clear liquid through a filter, add 100 cc. of boiling water to the precipitate, let it settle and then pour off the clear liquid through the filter, repeating the process till the filtrate is free from chlorides; finally transfer the precipitate to the filter, wash this with distilled water, dry the precipitate and transfer it to a tared crucible, separating the precipitate from the filter as completely as possible, burn the filter separately, letting the ash fall into the crucible, heat this to low redness, cool, and weigh. Subtract the filter ash from the increase in weight, and multiply the remainder by 0.34335 for SO_3 in 2 grs. of ash. (Preserve the filtrate and washings for (11) *Estimation of alkalies.*)

8. *Oxide of iron.*—Filter 100 cc. of the original acid solution for (7) through a dry filter, nearly neutralize with ammonia water, then add 1 gr. of sodic acetate and acetic acid till the odor of acetic acid is perceptible, boil to precipitate ferric phosphate, filter while hot, and wash the precipitate with boiling distilled water till the filtrate is free from chlorides. Dissolve the ferric precipitate on the filter with dilute H_2SO_4 into a small Erlenmeyer flask, wash the filtrate, dry, and ignite the same, and add the ashes to the acid solution in the flask, reduce the ferric to ferrous salt by amalgamated zinc or by a coil of magnesium wire, till a drop of the solution gives no color, with NH_4CyS. Pour off the solution of ferrous salt into a beaker, rinse the flask and add the rinsings to the beaker, add freshly boiled distilled water to make 200 cc. of the solution, add 2 cc. of sulphuric acid, heat to 70° C., and titrate with standard solution of permanganate, and estimate the iron as ferric oxide.

9. *Lime.*—Evaporate the filtrate and washings from ferric phosphate (8) to 100 cc. To the hot solution add 20 cc. of concentrated solution of ammonic chloride and 40 cc. of saturated solution of ammonic oxalate; boil the whole for ten minutes, and then let it stand in a warm place for six hours; decant the clear liquid upon a filter, wash the precipitate twice by decantation, then bring the precipitate upon the filter and wash it free from chlorides and oxalates, testing the washings by argentic nitrate. Puncture the point of the filter with a glass rod, wash the calcic oxalate into a 500 cc. flask by a stream of the wash bottle, dissolve any oxalate adhering to the filter by dilute H_2SO_4 (1:10), add 20 cc. of H_2SO_4 to the flask and make the volume to 300 cc. with pure water, heat to 70° C. and titrate with a standard solution of permanganate in which each cubic centimeter is equivalent to 0.0063 gr. of crystallized oxalic acid. Each cubic centimeter of the permanganate solution used will then be equivalent to 0.0028 gr. of CaO.

Alternate method for lime.—Dry the washed precipitate of calcic oxalate; transfer the oxalate to a tared crucible, burn the filter on a platinum wire, letting the ashes fall into the crucible, heat the crucible to a low red heat, cool, and moisten the contents with a saturated solution of ammonic carbonate, dry and heat carefully to a low red heat to expel ammonia salts, cool, and weigh. Subtract the weight of filter ash, and estimate the increased weight as $CaCO_3$.

Second alternate method for lime.—Dry the washed precipitate of calcic oxalate, transfer to a tared platinum crucible, burn the filter, and add the ash to the contents

of the crucible. Overflow the calcic oxalate with conc. H_2SO_4, heat gently to dryness, and then intensely to expel excess of H_2SO_4, cool in desiccator and weigh. The increase of weight, minus filter ash, estimate as calcic sulphate, $CaSO_4 \times 0.41158 = CaO$.

10. *Magnesia.*—Evaporate the filtrate and washings from calcic oxalate to 200 cc., pour into a clean and unscratched Erlenmeyer flask of 500 cc. capacity, add 30 cc. of a strong solution of $(Na)_2HPO_4$ and 50 cc. of conc. ammonic hydrate, cork the flask and shake violently at intervals of a few minutes till crystallization is established, and then set aside for twelve hours in a cold place. When precipitated in this way crystals will seldom adhere to the sides of the flask, yet more perfect crystallization is obtained than when stirred with a glass rod. If crystals should form on the sides of the flask they are as readily detached by a rubber-tipped rod as in a beaker. Filter through a tared Gooch filter, wash the precipitate with ammonic hydrate, diluted with distilled water (1 to 3), till the filtrate is free from phosphates (acidify a few drops of the filtrate with nitric acid and test with molybdate of ammonia). Dry the precipitate, ignite, at first very gently and then intensely, with a blast lamp, to convert $MgNH_4PO_4$ into $Mg_2P_2O_7$. Cool in a desiccator and weigh. The increase of weight $\times 0.36024 = MgO$ in 2 grs. of air-dry ash.

11. *Estimation of alkalis.*[1]—Concentrate the filtrate and washings from (7) to 100 cc., add NH_4HO and $(NH_4)_2C_2O_4$ to complete precipitation of barium and calcium, filter, wash the precipitate, evaporate the filtrate and washings to dryness in a platinum dish and ignite gently. Add to the residue concentrated solution of oxalate of ammonia, evaporate to dryness and ignite gently. Dissolve the residue in distilled water, filter from insoluble MgO, acidify the filtrate with HCl, and evaporate to dryness in a tared platinum dish and ignite gently. The increase of weight represents the chlorides of potassium and sodium in 2 grs. of ash. Separate and estimate potassium by $PtCl_4$, in the usual way, and the sodium by difference.

12. *Manganese.*—Manganese is not a usual constituent of wood ashes. Test the ash for manganese by heating on platinum foil over a colorless Bunsen flame 0.5 gr. of ash with 1 gr. of sodic carbonate and a few grains of nitrate of potash. The green manganate of soda in the fused portion as it cools will show the presence of manganese.

To estimate manganese, dissolve 2 grs. of ash in HCl. Evaporate the excess of acid over the water bath, pour the whole on a filter and wash with distilled water to make 100 cc., nearly neutralize with sodic carbonate, and then add 0.5 grs. of sodic acetate and 20 cc. of strong bromine water, and set the flask aside in a warm place for twenty-four hours, or until the bromine has nearly disappeared. Filter out the manganese oxide, wash thoroughly, transfer to a tared crucible, heat gently, and then intensely, and estimate the residue as Mn_3O_4. $Mn_3O_4 \times 0.93013 = MnO$.

The manganese may be precipitated by passing a stream of chlorine through the solution till fully saturated, instead of using bromine water.

The ash of mineral coal contains only a small amount of alkalis and phosphates, but a large amount of insoluble material, clay, etc. The value depends mostly upon the sulphate of lime and phosphate present. They are often decomposed with difficulty. They should be ground to a fine powder, and 5 grs. placed in the digestion vial with 50 cc. of HCl (sp. gr. 1.115), and digested in the steam bath for six hours, and the soluble portion analyzed in the usual way.

The aluminic material is in so large proportion that it is better to use the molybdic method for estimating phosphoric acid after eliminating soluble silica.

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[1] Test the filtrate from (7) for lithia by the spectroscope with a loop of platinum wire moistened with the filtrate held in a colorless Bunsen flame. The quantity of lithia is usually too small to be determined gravimetrically, but it may be estimated by diluting the solution with distilled water till the lithia line is on the point of disappearing from the spectrum, when a loop of clean platinum wire moistened with the solution is placed in the Bunsen flame. The wire must be clean for each trial, and no concentration of the salt should take place by repeatedly evaporating the solution on the loop of wire without cleaning it. One part of LiCl in 450,000 parts of water will show the lithia line in the spectrum.

TABLE OF ATOMIC WEIGHTS.

[Issued December 6, 1890.]

Revised for the Committee of Revision and Publication of the Pharmacopœia of the United States of America, and adopted by the Association of Official Agricultural Chemists.

By F. W. CLARKE, *Chief Chemist of the United States Geological Survey.*

[This table represents the latest and most trustworthy results, reduced to a uniform basis of comparison, with oxygen = 16 as starting point of the system. No decimal places representing large uncertainties are used. When values vary, with equal probability on both sides, so far as our present knowledge goes, as in the case of cadmium (111.8 and 112.2), the mean value is given in the table.]

[The names of elements occurring in pharmacopœial medicinal chemicals are printed in heavy-faced type.]

Name.	Symbol.	Atomic weight.	Name.	Symbol.	Atomic weight.
Aluminium	Al	27	Molybdenum	Mo	96
Antimony	Sb	120	Nickel	Ni	58.7
Arsenic	As	75	Nitrogen	N	14.03
Barium	Ba	137	Osmium	Os	190.8
Bismuth	Bi	208.9	Oxygen[4]	O	16
Boron	B	11	Palladium	Pd	106.6
Bromine	Br	79.95	Phosphorus	P	31
Cadmium	Cd	112	Platinum	Pt	195
Cæsium	Cs	132.9	Potassium	K	39.11
Calcium	Ca	40	Rhodium	Rh	103
Carbon	C	12	Rubidium	Rb	85.5
Cerium	Ce	140.2	Ruthenium	Ru	101.6
Chlorine	Cl	35.45	Samarium	Sm	150
Chromium	Cr	52.1	Scandium	Sc	44
Cobalt	Co	59	Selenium	Se	79
Columbium[1]	Cb	94	Silicon	Si	28.4
Copper	Cu	63.6	Silver	Ag	107.92
Didymium[2]	Di	142.3	Sodium	Na	23.05
Erbium	Er	166.3	Strontium	Sr	87.6
Fluorine	F	19	Sulphur	S	32.06
Gallium	Ga	69	Tantalum	Ta	182.6
Germanium	Ge	72.3	Tellurium	Te	125
Glucinum[3]	Gl	9	Terbium	Tb	159.5
Gold	Au	197.3	Thallium	Tl	204.18
Hydrogen	H	1.007	Thorium	Th	232.6
Indium	In	113.7	Tin	Sn	119
Iodine	I	126.85	Titanium	Ti	48
Iridium	Ir	193.1	Tungsten	W	184
Iron	Fe	56	Uranium	U	239.6
Lanthanum	La	138 2	Vanadium	V	51.4
Lead	Pb	206.95	Ytterbium	Yb	173
Lithium	Li	7.02	Yttrium	Yt	89.1
Magnesium	Mg	24.3	Zinc	Zn	65.3
Manganese	Mn	55	Zirconium	Zr	90.6
Mercury	Hg	200			

[1] Has priority over Niobium.
[2] Now split into Neo- and Praseo-Didymium.
[3] Has priority over Beryllium.
[4] Standard or basis of the system.

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